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OSRD No.

5626

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DIVISION 8
NATIONAL DEFENSE RESEARCH COMMITTEE
of the
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THE PREPARATION AND PROPERTIES OF RDX-COMPOSITION A

Explosives Research Laboratory

OSRD Report No. 5626

Copy No. 65

Date: December 29, 1945

Service Projects: NO-290, OD-01

Endorsement from Dr. Ralph Connor, Chief, Division 8 to Dr. Irvin Stewart, Executive Secretary of the National Defense Research Committee. Forwarding report and noting:

"One of the first problems studied at the Explosives Research Laboratory (in June 1941) was that of finding a suitable substitute for the beeswax used by the British in their RDX/Wax Semi-plastic Explosive. The results achieved in this study up to February 1942 are contained in two earlier reports, OSRD-313 and 397. By that time, the specific objective of finding a suitable wax or waxes to be used in the same way the British used beeswax had been attained. The present report deals with the study of various physical properties of Composition A, and with work on a number of problems which arose in the course of putting this material into production and service use.

During the original investigation it was realized that a good desensitizer for RDX should probably have physical properties similar to those of beeswax or petroleum waxes. However, the belief was held that the desensitizing effectiveness of waxes might be highly specific; that is, some waxes might be good desensitizers, while others which did not differ from them in any major way might be poor. The point of view held by the authors of the present report is that any petroleum wax having a hardness within a fairly broad range will be an effective desensitizer. Consequently the choice of a wax for use in making Composition A should be based on considerations of supply, melting point, exudation, and, of course, consistency. It still remains true, as was pointed out in the first report, that most neutral petroleum waxes are not taken up well by RDX from a hot water slurry, and consequently it is necessary to add to the wax some material having polar groups. The first material recommended for this purpose, Alox 600, remains a good choice, but it has been found that 1%, rather than the 10% previously suggested, is adequate. Cutting down the amount of
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Alox is an advantage, as it helps to reduce exudation from the Composition A during hot storage, and it also makes the explosive less likely to stick to tools and dies when it is pressed.

Anyone interested in the problem of writing specifications for waxes should also consult OERD-5633 which deals with the problem of evaluating waxes for use as desensitizers in various compositions, including Composition A."

This is a progress report under Contract OEdsr-202 with the Carnegie Institute of Technology.

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Progress Report

OSRD No. 5626

on

**The Preparation and Properties of
RDX-Composition A**

from the

**Explosives Research Laboratory
Bruceton, Pennsylvania**

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Approved by:

D. P. MacDougall
December 6, 1945

Summary

This report reviews the studies of Composition A made in this laboratory since February 15, 1942; previous work has been reported in OSRD Reports No. 313 and 397. Preparation of Composition A is first discussed. Of the possible methods of coating RDX with wax, the slurry method is deemed best, but to most neutral petroleum waxes wetting agents like Alox 600 must be added to secure good coating of the crystals from water. Roll-milling of the wet product produces a material which is plastic when heated above the melting-point of the wax, but does not improve the desensitizing action of the wax if coating of the crystals has been good. This hot plasticity is not required in American loading practice, and moreover, the tray-dried unrolled product (Composition A-3) has a desirable granulation without any special graining process.

Next, a thorough small-scale study of the pelleting properties of Composition A is presented. It is shown that the pellet-density may be represented basically as a function of the wax

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hardness (measured by the ASTM Penetration Test) and the pressure. Effects of pellet diameter, pellet length, pressing time, and even of complicated increment pressing schedules may be interpreted in terms of the basic pressure-penetration-density relations. With Composition A-3 rather similar results are obtained. The Explosive properties of Composition A are next discussed. It is first shown that among the petroleum waxes which are suitable for use in this product, from the standpoint pelleting properties, each seems to be equivalent as a desensitizer, if well applied as a coating. Composition A, because of its constitution and mechanical properties, is not easy to compare, in sensitivity, with pure-compound explosive materials. Most drop-weight tests show it to be more sensitive than TNT, while the Bullet-Impact and Booster Sensitivity Tests show it to be less sensitive than TNT (in the same physical form, of course). Considered generally, small scale tests place Composition A on the sensitivity scale as clearly less sensitive than Tetryl and more sensitive than Ammonium Picrate-probably in the same class as TNT (pressed). Small-scale tests of performance, on the other hand, show that this material appears to outperform not only TNT but also Tetryl in every respect which has been investigated.

The chemical stability of Composition A's made with neutral petroleum waxes appears to be essentially as high as that of RDX itself; certain synthetic waxes, however, give rather unstable products. The loss of wax in high-temperature storage (60°C) depends upon the wax used, and among waxes having melting points between 160 and 175°F. may range from 0.4% to 15% of the wax in five weeks. Finally, such variants of Composition A as Aluminized Composition A, 12% Composition A, and Composition A-KNO₃ mixtures are briefly discussed or referred to.

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Report

1. Introduction

In OSRD Report No. 313, work at this laboratory directed toward discovery of a substitute for beeswax for the phlegmatization of RDX has been reviewed up to Dec. 30, 1941. OSRD Report No. 397 completed the description of this work through February 15, 1942. Of the ten waxes suggested in these reports, one was selected for use in RDX Compositions A and B, and has so been used throughout the war. With this adoption of 90/10 Aristowax 160-65- Alox 600, which, since it was the tenth wax listed, has been called "Bruceton Wax No. 10", one phase of our work with Composition A came to a close. Pilot operations were begun at the Eastern Laboratory of the duPont Co., and finally full-scale production began at the Wabash River Ordnance Works. Thus, after about the summer of 1942, problems concerning Composition A have come to us more or less as a few direct requests. In this way we were requested to study the first samples of unrolled Composition A (Comp. A-3) from the duPont Co., to evaluate certain waxes as possible substitutes for Aristowax 160-65, to suggest possible substitutes for Alox-600, or to seek waxes whose use in Composition A would further reduce exudation. The basic reason why so few problems concerning Composition A have emerged lies, to be sure, in the fact that this explosive has not been used by the services until this year, when it has been loaded by the U.S. Navy in the 5"-38 A.A. Shell; this development came, however, too late for the clear-cut definition of, or a completed laboratory attack upon real problems developed in use.

Nevertheless, during the period since February 1942 a good deal of work has been done with Composition A in this laboratory, partly on request, and partly simply in the course of general investigations of desensitization and properties of explosives. This work, coupled with the growth of experience at the E.R.L., has gradually produced so many changes in our point of view that the underlying tone of the two early reports is now no longer fairly representative of our beliefs, though the facts presented are generally true, and the wax which was chosen from the recommended list in O.S.R.D. No. 313 has proved most satisfactory (except for changes in the properties of the Aristowax in production over a period of time, a matter which could scarcely have been foreseen, and which has had no demonstrable ill effects in any case). Accordingly we shall in this report summarize the work done with Composition A at E.R.L. since February 15, 1942, and stress changes in opinions and points of view which have arisen during that period.

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2. Studies of the Preparation of Composition A.

(1) Application of Wax to RDX

It has been generally accepted that the most satisfactory method of incorporating the wax with the RDX is by the addition of molten wax to a well-agitated water slurry of RDX, held above the melting-point of the wax. In the laboratory, with batches varying from 50g. up to 10 lbs., we have slowly added molten wax to a rapidly stirred slurry of RDX in water (1:3, RDX: water) at about 90°C. When a wax suited to this process is employed, the RDX crystals are completely coated with wax after 5-6 minutes of stirring and have agglomerated to small round grains which, when stirring is stopped, settle rapidly to the bottom of the kettle. The slurry of grains may then either be allowed to cool with stirring, or, if the batch is large, be cooled rapidly by the addition of cold water to the kettle. When cool, most of the water may be decanted, and the residue removed on a filter. Commercial practise generally does not deviate in principle from this procedure. We have made numerous laboratory studies of the exact conditions which give a well-coated granular product of suitable grist, but such procedures are not necessarily applicable to large-scale production, so need not be detailed here. If too little water is used, the product does not ball up well to suitable grains; too much water, inadequate stirring, too high a viscosity of the molten wax, and too low an oiling temperature slow down the rate at which RDX crystals are coated.

a. Coating Properties of Waxes

Although beeswax is suited to the process outlined above, neutral petroleum-base waxes in general are not, although some highly colored and impure amorphous waxes do appear capable of wetting RDX from water in this manner. On the other hand, most neutral petroleum-base waxes are taken up by RDX in such a slurry-mixing, and do not remain floating on the water layer when agitation has stopped. When such waxes are colorless, it is hard to be sure, on casual inspection, that the RDX has not been coated (though the uncoated product never grains, but remains fine). The ultraviolet examination technique developed at Picatinny Arsenal^{1/} permits an easy differentiation between RDX samples which simply contain wax, and samples which have been well coated. Plate 1 shows photomicrographs at 4.6 x of samples of RDX, of RDX / 9% Aristowax, and of RDX / 9% Aristowax-Alox (Bruceton Wax No. 10) viewed in ultraviolet fluorescent light, and in white light. Each waxed sample was made by the slurry method, and in each case the wax was apparently taken up by the slurry. The difference between the coarse grains of the coated RDX and the fine, uncoated material is, of course, apparent, but the

^{1/} Technical Report, Picatinny Arsenal, Serial No. 1487, January 17, 1945. "Desensitization of High Explosives by Waxes"

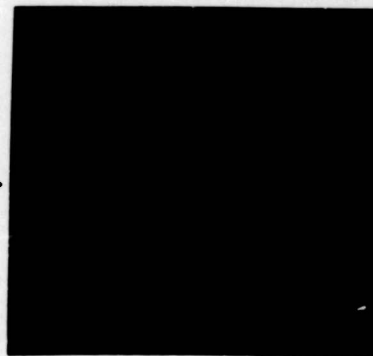
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White Light

Fluorescent Light



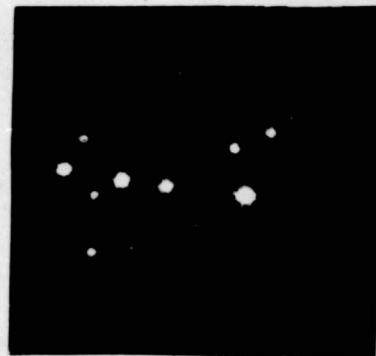
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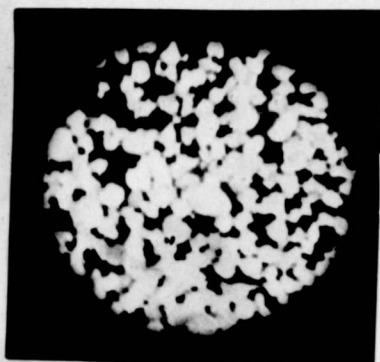
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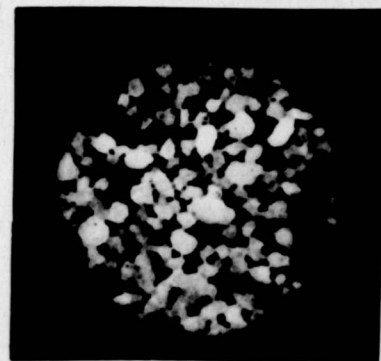
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RDX plus 9% Aristowax



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RDX plus 9% Aristowax-Alox (Bruceton Wax No.10)

Plate 1

Magnification: 4.6x

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photographs in fluorescent light reveal clearly that the wax in the uncoated sample is actually present only in a few globules, while the sample made with Alox is uniformly coated. Now if the filtered product is to be subjected to a series of roll-millings, (Composition A) failure to coat completely might not be objectionable, but if the product is to be tray-dried and used directly (Composition A-3), failure to coat the RDX well will give very sensitive products. The three samples shown in Plate 1 gave, for example, when tested as a group on the Type 12 Impact Machine,^{2/} the following 50% points in centimeters:

| <u>Sample</u> | <u>50% Point (cm.), Type 12</u> |
|----------------------|---------------------------------|
| RDX | 15 |
| Comp. A-3, no Alox | 31 |
| Comp. A-3, with Alox | 72 |

Bullet tests, using samples pressed to a density of 1.0 in 1" pipe-nipples, and Cal. 0.30, Ball, M2 Ammunition, gave high-order detonations in each of ten trials with a Composition A-3 made without Alox, while 59 samples of well-coated Composition A-3 have each been inert under the same test conditions.^{3/ 4/} It is certain that the suggested waxes numbered 7 and 9 in OSRD Report No. 313 do not coat RDX from water, and it is probable that the waxes numbered 1 and 3 do not coat well.

^{2/} O.S.R.D. Report No. 5744

^{3/} O.S.R.D. No. 5745.

^{4/} Picatinny Arsenal Technical Reports 1489 and 1499 present similar and more detailed correlations between wax coverage and sensitivity for PETN-wax systems.

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It now seems clear that most petroleum-base waxes form in water an interfacial contact angle on RDX crystal surfaces which is finite; accordingly RDX particles adhere to wax globules and retain them in the slurry. The RDX is neither capable of acting as an efficient powder-emulsifier for wax in water nor is it, on the other hand, completely wet and coated by wax. To assure the latter behavior, which is shown by beeswax (and certain petroleum waxes which presumably contain polar impurities adequate as wetting agents), it is generally required that a wetting-agent be added to the wax to make the wax-water contact angle on RDX zero. For this purpose the catalytically oxidized petroleum product called Alox 600 (Alox Corporation, Niagara Falls, N.Y.) was suggested in OSRD Report No. 313 (1941), and was adopted (as an ingredient of Wax #10). This additive has been satisfactory, in the main, but has presented two unrelated difficulties. Alox samples generally contain small quantities of resinous material insoluble in molten wax, in amounts which vary somewhat from sample to sample. This dark material is sticky and possibly troublesome in production; it moreover appears as dark flocks in the product, which perhaps tend to spoil its appearance. No real harmful effects have ever been identified with this resin. It has been more recently observed that the recommended concentration (10%) of Alox 600 in the WAX #10 is considerably more than is needed to ensure good coating, and that the Alox tends to increase stickiness and wax exudation. It is recommended that a reduction of the Alox 600 content from 10 to 1% be made, to reduce exudation and stickiness.

Since Wax #10 was also approved for use in Composition B, considerable quantities of Alox 600 were used during the war, and at one time requests were received to make recommendations for a substitute for Alox 600. Several materials, including Stearic Acid, cetyl alcohol, and anhydrous lanolin were tried. Of these it was felt that lanolin most nearly resembled Alox in its effects. Stearic Acid hardened the base wax considerably, and tended to make the product discolor in contact with brass. Cetyl alcohol increased the exudation more than Alox 600. It was not actually necessary to employ any substitute for Alox 600. O.S.R.D. Report No. 313, p.6, lists a large number of other additives which have been tried, especially with paraffin.^{2/}

5/ Picatinny Arsenal Technical Report 1533 presents information concerning a somewhat different application of wetting agents in PETN-wax systems to secure uniform coatings and uniform particle sizes in final products.

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b. The Effects of Wax Viscosity

A few waxes have been tried in Composition A which, despite the addition of 10% Alox 600, failed to give good coating from water except with unusually vigorous stirring and at unusually high temperatures. Two such waxes were B-square Wax and the tank-bottom wax Super Gem Amber. It is believed that these amorphous waxes had too high a viscosity at the oiling temperature and were, accordingly, being prevented from spreading by purely mechanical means. The following viscosities were determined at 210°F with the Brookfield Viscometer:

| <u>Wax</u> | <u>Viscosity (centipoises)</u> |
|---------------------|--------------------------------|
| Super Gem Amber | 16.0 |
| B-Square Wax | 12.3 |
| Superla Wax, Yellow | 10.4 |
| Stanolind, 170/75 | 8.5 |
| Aristowax, 160/65 | 7.4 |
| Stanolind, 160/65 | 6.2 |
| 50° Paraffin | 4.3 |

It is known that Stanolind 170/75 wax oils well at reasonable temperatures, and that Superla Wax Yellow is fairly satisfactory; accordingly, it seems wise to use waxes whose viscosity is below about 9 centipoises, or below about 60 Saybolt Universal Seconds at 210°F.^{6/} An allied difficulty has been encountered in attempting to prepare samples of waxed RDX containing 5% of wax. Under these circumstances, the wax does not seem to spread evenly in a thin coat over all the RDX. By adding not the molten wax, but a hot solution of wax in CCl₄, a smooth coating can be obtained, the solvent being evaporated off.^{7/} When too fine an RDX, such as the through 325 cut of a production RDX is used to make a regular Composition A, a similar difficulty is encountered, which also seems to be a mechanical one, rather than the result of the surface's being too great to be covered by the available wax (in this case the amount of wetting agent adequate for normal grist RDX may have become inadequate, however).

^{6/} It seems to us that the upper limit, 75 Saybolt Universal Seconds, allowed by Picatinny Arsenal Tentative Specification PKS-1127 is higher than desirable.

c. Other Methods of Coating

In early laboratory studies of wax desensitizations, certain waxes which could not be applied without additives from water, such as paraffin, were distributed mechanically over the RDX surface by dissolving the wax in CCl_4 , mixing the solution with dry RDX, and evaporating off the solvent as the sample was stirred. Judged by sensitivity tests, fair coatings were thus obtained, but the method seems not to be suited to largescale procedure, and can in any case be easily avoided by addition of wetting agents. Various methods of coating PETN with wax have been studied at Picatinny Arsenal.^{7/} Coprecipitation of PETN and wax from acetone by water addition, and co-crystallization of PETN and wax from ethyl alcohol, as well as coating in water slurry were tried. The latter method gave by far the best coatings.

(2) Rolling

a. The Effect of Rolling on Sensitivity

Early in the course of study of Composition A in these laboratories, it was believed that roll-milling of the wet waxed product was required to give the full desensitizing effect of added wax. Before differential rolls had been obtained, mechanical mortars were used to wet-grind waxed samples. At this time sensitivity tests were made with the Type 4 Impact Machine, and the ratios of the measured 50% points for the waxed sample and for RDX were quoted as "desensitization ratios". With this mortar-grinding technique and this Impact Machine effects of milling on the desensitization ratio were studied. Results such as these are typical: One hour's wet milling of a beeswax coated sample raised the desensitization ratio from 1.9 to 2.8; milling (of unspecified duration) increased the desensitization ratio of a paraffin-stearic acid coated sample from 2.7 to 7.6. Results of this sort temporarily convinced workers that somekind of milling was required for good desensitization. When differential rolls were obtained the following milling series was studied; rolling of the Beeswax-Coated RDX was at 0.005" roll separation, with unsprung rolls (i.e. infinite spring force).

| <u>Number of Passes</u> | <u>50% Pt. (cm); Type 4.</u> |
|-------------------------|------------------------------|
| 1 | 65 |
| 5 | 95 |
| 7 | 74 |
| 9 | 108 |
| 11 | 0% at 115 cm. |

^{7/} Picatinny Arsenal Technical Report 1489.

^{8/} Sec O.S.R.D. Report No. 5744.

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These results appeared to confirm the earlier ones. In December of 1942 a sample of grained Composition A and of Composition A-3 (unrolled), made by the duPont Co. from Bruceton Wax No. 10, were received for comparison. At this time the Type 4 machine had been abandoned as too irreproducible, so the samples were tested on the Type 7 machine (See footnote 8), giving the following results:

| <u>Explosive</u> | <u>50% Point (cm); Type 7</u> |
|----------------------------|-------------------------------|
| Composition A | 151 |
| Composition A-3 (unrolled) | 150 |
| TNT (average of 10 trials) | 153 |

Bullet tests of these two materials hand-tamped in 1" pipe-nipples, tested with Cal. 0.30, Ball, M2 ammunition also revealed no sensitivity difference. Accordingly a new milling series was made, using sprung rolls at 0.020" roll separation and 460 lb. spring-force (which was then current laboratory practice), and also unsprung rolls and a 0.005" roll separation, as was used in the earlier series. The following results were obtained.

| <u>Passes</u> | <u>Roll Separation</u> | <u>Spring-Force</u> | <u>50% Point (cm) Type 7</u> |
|---------------|------------------------|---------------------|------------------------------|
| None | -- | -- | 110 |
| 2 | 0.020" | 460 lb. | 117 |
| 8 | 0.020" | 460 lb. | 115 |
| 2 | 0.005" | ∞ | 86 |
| 8 | 0.005" | ∞ | 95 |

Four bullet-tests under the conditions already mentioned were made on each sample, and no detonations were obtained. Taken at face value, these results suggest that the unrolled material was well covered, and its sensitivity was not affected by milling with sprung rolls at a rather wide separation, but that milling with close, unsprung rolls actually fractured coated crystals, and only by repeated passes was the coating re-established. To test this notion, a well coated sample was milled one pass through unsprung rolls at 0.001" separation and tested at density 1.0 in the Cal. 0.30, Ball, M2 bullet test, using 1" pipe nipples. Results were:

| <u>Explosive</u> | <u>No. Trials</u> | <u>Percent Inert</u> |
|-------------------------|-------------------|----------------------|
| Comp. A-3 (unrolled) | 59 | 100% |
| Comp. A-rolled one pass | 5 | 80% |

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Few trials were made with this violently rolled material, but it seems to be more sensitive than the unrolled product. Since this time, rolled and unrolled samples have been compared on several occasions with the Type 12 Impact Machine (See reference 8); no difference has been detected. The early results seem, therefore, to have been misleading for reasons which cannot be reconstructed in detail. It is possible that in early small scale preparations, made with hand stirring the coatings of wax were not very good, and that with those samples (as with any badly coated ones) the mechanical effects of rolling or grinding of suitable violence were helpful. The early milling series did not include a test of the unmilled product, so that perhaps it reveals only the effects of re-covering, by milling, of surfaces exposed by the first pass through the rolls. In any case, no evidence now exists to assert that a well-rolled and a well-coated unrolled Composition A display different sensitivities. On the other hand, it is certain that a poorly coated unrolled material will be unduly sensitive, and it is probable that a few passes of a well-coated material through close, unsprung rolls will increase rather than decrease its sensitivity.

b. The Effects of Rolling on the Plastic Properties of Composition A.

If Composition A and A-3 are equivalent in sensitivity, the former displays markedly superior plastic properties when warmed above the melting point of the wax. This difference is the result of the favorable particle size distribution of the RDX produced by roll-milling, and is a difference which is of great importance when Composition A is to be loaded as a semiplastic by hot stemming, which is British loading practise with this material, but which has never been seriously contemplated in this country. This effect of rolling became apparent to us only when our first sample of British product was received early in 1942. At this time also a few of the details of British rolling practise were received, and over a series of months we investigated rolling procedures in an attempt to match the plastic properties of the British product. When the warmed British material was pressed into thin plates and cooled, these plates were characterized by considerable hardness; such plates break with

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a sharp snap, and when viewed under the microscope are found to be well consolidated and close grained. We were easily able to avoid the dry powdery products produced in early rollings, but the best samples crumbled rather than snapped, and were always found under the microscope to be of rather open grain. Finally, in December of 1942 the first Composition A samples from the larger rolls at the Eastern Laboratory of the duPont Co. were obtained; these seemed comparable in hot plasticity and cold hardness to the British samples. Shortly thereafter it appeared that the rolled product would not find use in this country, so further attempts to discover the correct conditions for small-scale milling were abandoned.

Nevertheless, there are conditions under which a good rolled product is very useful. One of these is in the production of the British pourable RDX-Wax Aluminum mixture, which is prepared by the addition of extra wax and aluminum, plus lecithin, to a rolled Composition A. With a total of 12% wax, such a mixture can be poured, but with unrolled Composition A one needs almost 20% of wax.

(3) Drying

British products and Composition A and A-2 as specified in this country were dried in a jacketed kettle equipped with an anchor-stirrer. In the laboratory we used a steam heated Banbury mixer, capable of drying a two-pound batch. These procedures produce coarse, lumpy products, hard when the material has been rolled, and more friable when it is unrolled or poorly rolled. Composition A-3 is tray-dried in a current of warm air at 150-160°F. In the laboratory we have dried this material in a forced-draft oven at 150°F. The maximum water content permitted by U.S. Army Ordnance Dept. Tentative Specification AXS-768 (Ref. 2), Feb. 7, 1945, is 0.1%. No particular investigation of the drying process has been made in this laboratory.

(4) Graining

At one time small-scale processes for graining the coarse lumps of Composition A in the differential rolls were studied and employed in the laboratory to produce products suited to pelleting. Procedures for glazing with graphite in a tumbling barrel were likewise used. These have become generally of no interest, since the Composition A-3 is directly

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obtained in a form suitable for pelleting, and graphite coatings, if desired, may be obtained directly by adding graphite, wet with water, to the slurry after the granules of Composition A-3 have formed; continued stirring coats the granules with graphite.

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3. Studies of the Pressing Characteristic of Composition A

When it became obvious that Composition A loading by the British hot-stemming method would not be acceptable in this country, it was felt that a thorough small-scale study of the pressing characteristics of Composition A should be undertaken. It was hoped that such a study might at once serve as a guide in choosing waxes which would produce products suited to any specific loading conditions, and constitute an aid in designing loading equipment and procedures for any specific Composition A. When this work was completed in the summer of 1942, no one seemed interested in loading Composition A, so the results have never been reported in a formal Progress Report; accordingly they will be presented now.

(1) The Variables; their Measurement or Control

a Internal Variables (Those Fixed for a Specific Composition A)

(a) Wax Content

All the samples of Composition A made in this laboratory were prepared with 9% wax by weight, and no attempt was made to vary this amount, although the sample of British material, which has also been studied, has been found to contain 9.40% wax.

(b) Wax Density

The densities of the various waxes used in this work were found to be sufficiently different to influence the measured results; accordingly the densities of all pressed charges have been corrected to the densities which would have been measured had the wax possessed the density of Bruceton Wax No. 10. This correction is large only in the case of samples made with beeswax, but is appreciable in other cases also. Within the density range of interest, this adjustment may be simply made by adding a correction to the measured density. The following Table gives the wax densities and additive corrections for the waxes studied:

| <u>Wax</u> | <u>Density</u> | <u>Correction</u> |
|---|----------------|-------------------|
| 1. Suprala Wax, Yellow | g/cc 0.936 | -0.002 |
| 2. Yellow African Beeswax | 0.958 | -0.008 |
| 3. Bruceton Wax No. 10 (90% Aristowax 160-65, 10% Alox 600) | 0.929 | 0.000 |
| 4. 90% Petrocene C, 10% Alox 600 | 0.933 | -0.001 |
| 5. 95% Gulf Petrowax .., 5% Alox 600 | 0.917 | +0.003 |

(c) Size Distribution of the RDX

All samples made in this laboratory were prepared from RDX having the same initial size distribution, and all were passed six times through a differential roll-mill at roll-separation 0.020"; accordingly it is hoped that this factor has been held constant. The British sample, however, has definitely a finer grist.

(d) Grain Size of the Grained Composition A

Both the British and our own samples were grained by the same procedure; furthermore control experiments have shown that the density of the pressed pellets is not very much altered even by large changes in the grist of the material pressed.

(e) Consistency of the Grained Composition

Grains of the British material were considerably harder and more compact than those of our products; the effects of this difference will be mentioned later. Among our own products, however, the consistency appeared to be a property which was independent of the specific wax used.

b External Variables (Those Fixed for a Specific Pressing)

(a) Pressure

The oil-pressure in the press was measured with an ordinary Bourdon pressure gage, calibrated against dead-weight pressure in a comparison apparatus. The pressure on the explosive was calculated in the usual way, the force required to float the plunger of the press being subtracted. It is felt that the maximum errors in the pressure were about $\pm 2\%$.

(b) Temperature

The molds were wrapped with heating elements, powered by a Varitrans, and the temperature of the mold was measured with a thermocouple deeply imbedded in the wall of the mold. In general, the explosive was loaded into the mold, pressed down firmly by hand, and then the mold was slowly brought up to temperature (in about 10 minutes), held there for about three minutes, and then the pressure was applied.

(c) Shape and Size of Charge

Most of our systematic work has been done with ten-gram pellets in a one-inch cylindrical mold. Variations in diameter and length of cylindrical charges, and variations to non-cylindrical shapes have been studied as well.

(d) Pressing Schedule

The effects of time of pressing, and of pressing in several increments have been studied.

(e) Others

The effects of tightness of the plungers and of the existence of paths of egress for entrapped air have been studied.

c. A Mixed Variable (The Wax Consistency)

It has proved useful to express the consistency of the wax in terms of the ASTM penetration test, Designation D5-25, employing a steel needle loaded with a 200 g. weight, and a time interval of 5 seconds. The results are expressed here in tenths of a millimeter penetration, and are designated by P (pressure will be designated p). This variable is fixed for a given Composition A only at constant temperature. Figure 1 gives the penetrations of four waxes chosen for study as functions of the temperature. It has been shown experimentally that two Composition A's made with different waxes give the same densities when pressed at the same pressures, provided the pressing temperatures are such that their penetrations are equal (at temperatures low enough so the waxes are not molten, of course). Accordingly, the identity of the wax and the temperature may be combined and expressed as one variable; the penetration, a device which is often useful and leads to great simplification of the results. The value of P, then depends both on the identity of the Composition A sample and on the temperature employed in any specific pressing.

(2) Preliminary Pressure--Density Isotherms

The curves of Figures 2-4 present the preliminary pressing data, obtained on ten-gram samples, pressed at various temperatures and pressures in a one-inch mold. The pellets generally were very smooth and regular when pushed from the mold, so that they could be measured very easily with a micrometer caliper; the diameters were very constant, and though there was some variation in the height of the pellet, it was generally sufficient to measure the height at the center and at four spots near the edges, and use the average height. Pellets were weighed on an analytical balance, and the densities computed directly.

a. The Effect of Melting of the Wax

One of the first striking features of these curves is demonstrated by the 75° curve of Figure 2, the only curve taken above the melting point of the wax. The slope of this curve at low pressures is much smaller than that of the other curves, and this slope persists much better at the high pressures, while the lower temperature curves flatten out and appear to reach a limiting density.

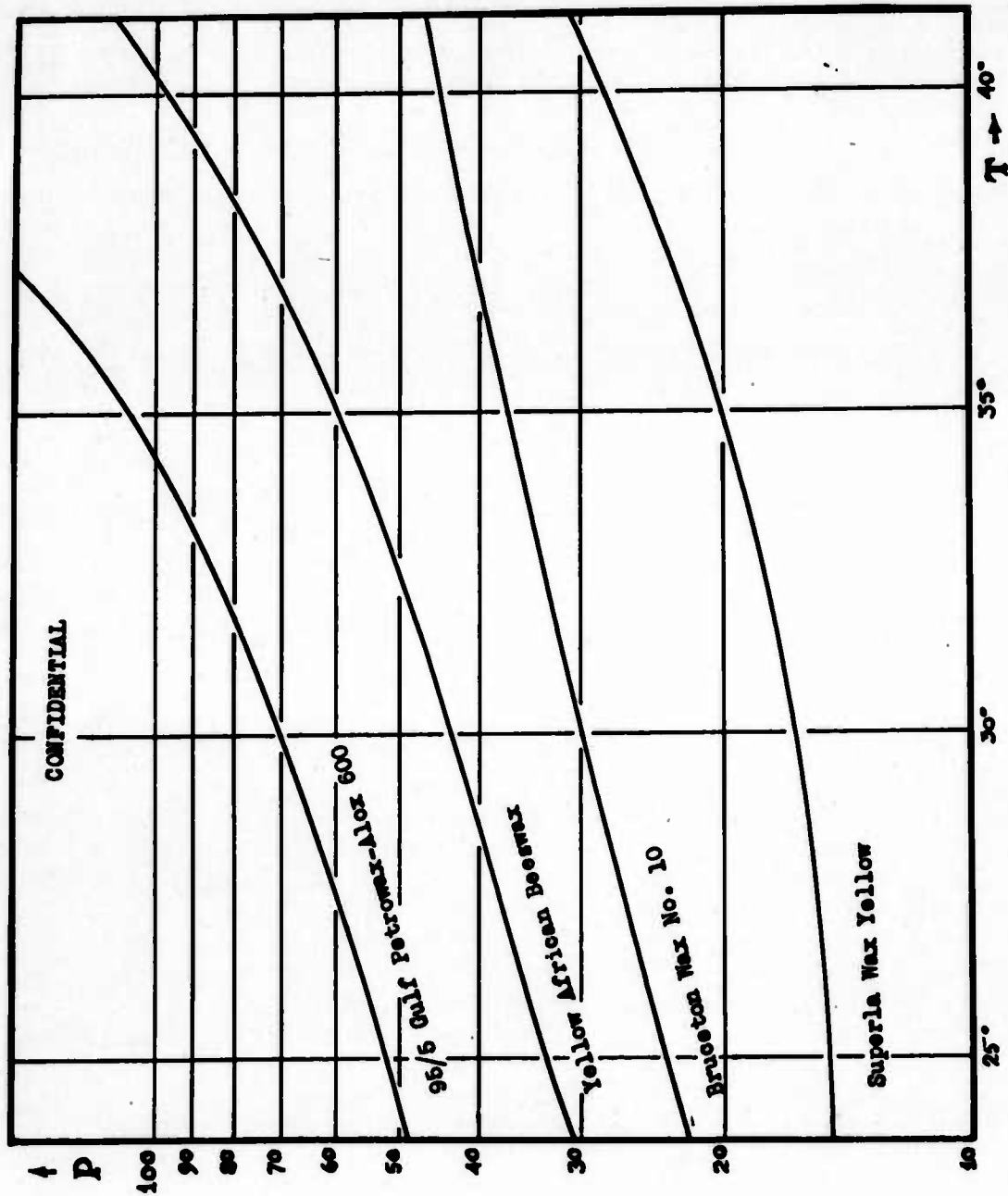


Figure 1. Penetration-Temperature Diagrams for Waxes Used in
Studies of Composition A.

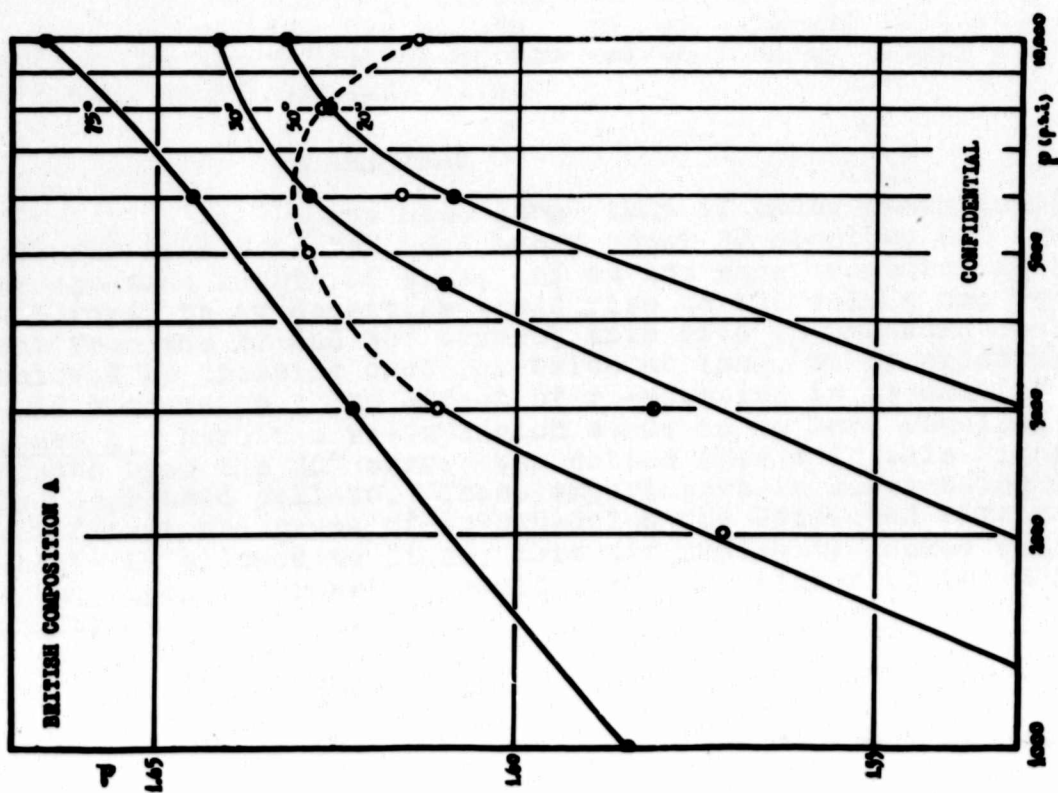


Figure 2. Pressure-Density Isotherms for British Composition A;
Ten-gran Pellets Pressed in a 1" Mold.

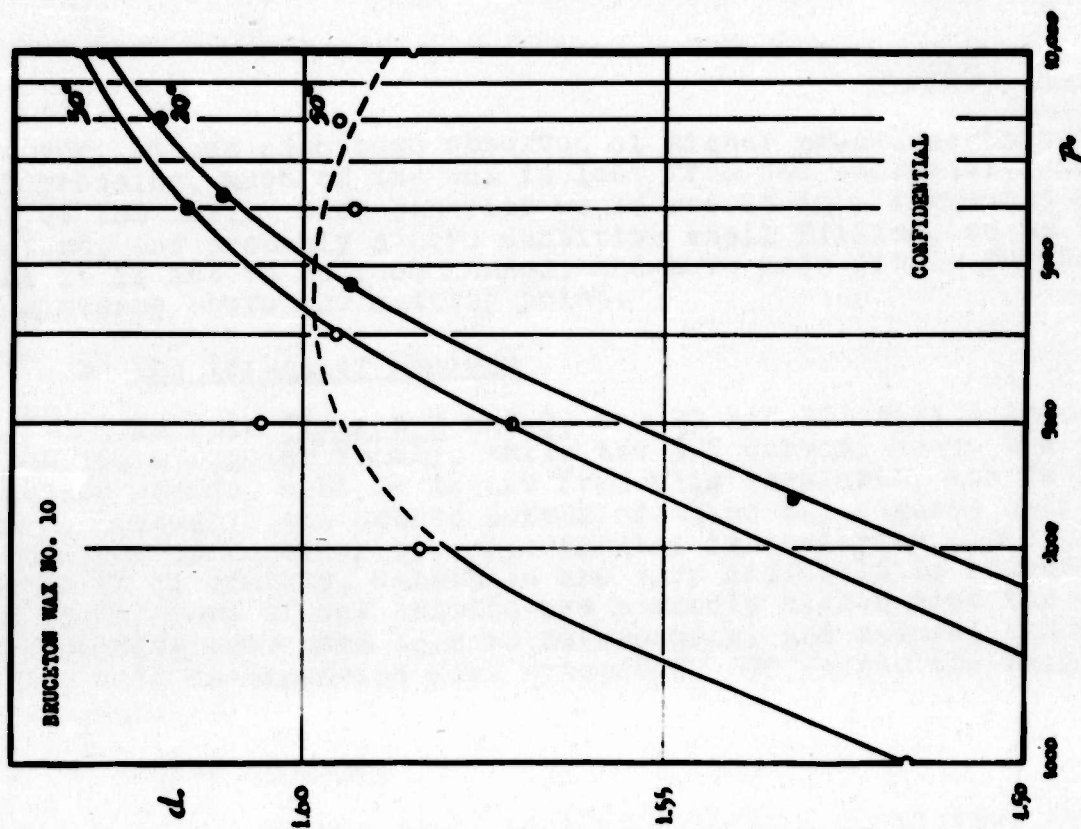


Figure 3. Pressure-Density Isotherms for Composition A Made
with Bruceston Wax No. 10; Ten-gran Pellets, 1" Mold

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However, it has also been observed at higher pressures that in this 75° pressing, much of the wax is lost from the explosive, and forced out of the mold. Such behavior would result in a decreased wax content, and probably a more sensitive shell filling, so we believe that it is not to be recommended, and have made little further study of mixtures above the melting point.

b The Irregular Regions

In both Figures 2 and 3, the 20 and 30° curves appear to belong to a regular family, while the 50° curves, above two or three thousand pounds, tend to depart from this regularity and fall off again. Actually the dotted curves of these two figures correspond to regions of considerable experimental irregularity also; pellets appear to be cracked, densities are very difficult to reproduce, and the centers of the pellets are markedly higher than the edges. Such pellets were said to have re-expanded, and several attempts to remedy this re-expansion were attempted. Of these, the following were successful:

(a) Cooling

It was found that if pellets were pressed at 50° for one minute, and then the mold cooled with ice as rapidly as possible, (this took about 15 minutes) with the pressure on, the cold pellet, when removed, showed none of the re-expansion effects, whereas a pellet held under pressure for the same length of time at 50° did exhibit the low density, cracks, and all the effects shown by the pellets pressed for one minute. It was believed that the effect of cooling was the hardening of the wax to such an extent that the re-expansion could not take place.

(b) Re-pressing

It was also found that if these re-expanded pellets were put back into the mold after about 30 minutes, and again pressed for the same length of time, and at the same pressure and temperature as before, their densities would rise to the values one would expect from the 20 and 30° curves; this rise in pressure could not be achieved by pressing once for twice as long, or by pressing twice in rapid succession. The effect of re-pressing is especially marked in Figure 4, where the re-expansion seems to be more regular, but affects even the 30° curve; the dotted curves in this Figure are for the re-pressed pellets. These experiments in re-pressing led to the belief that the cause of re-expansion was entrapped air; when the pellet was allowed to stand, this air gradually leaked out, and the pellet, upon re-pressing, would come much closer to its proper density.

(c) Vacuum Pressing

A mold was therefore prepared which enabled the

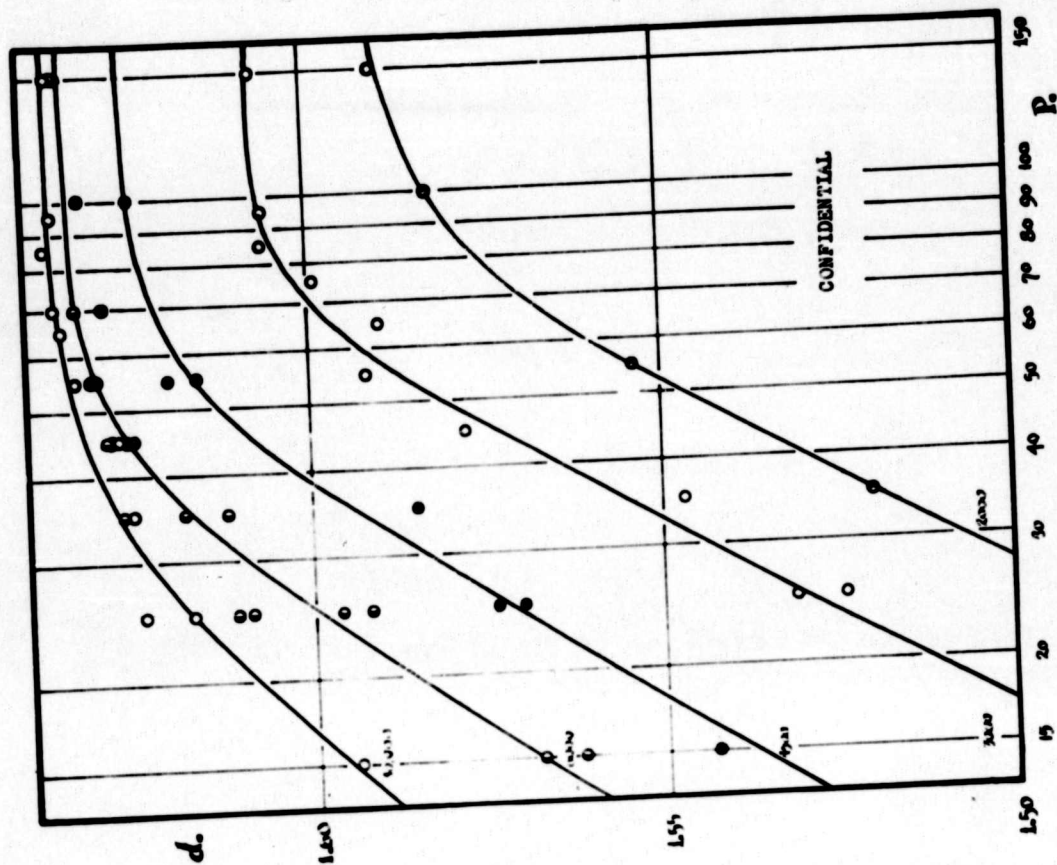


Figure 5. Penetration-Density Isobars for 1" Composition A Pellets; Ten-gram Pellets, Vacuum-Pressed.

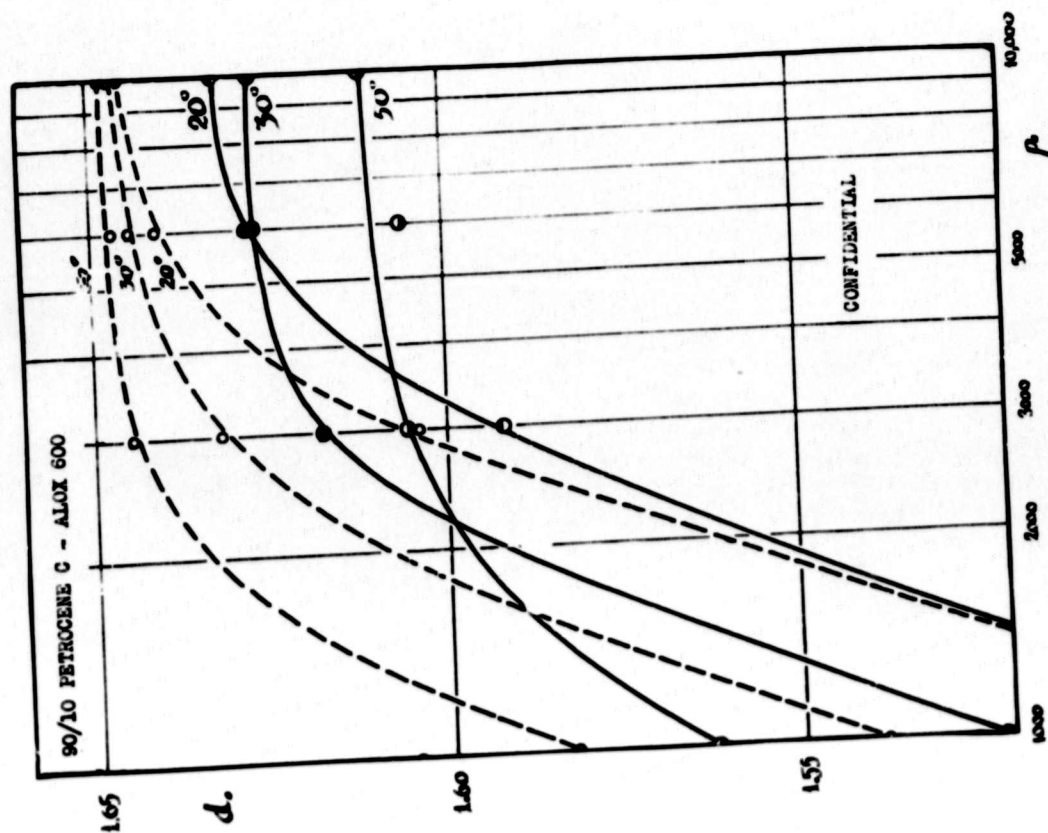


Figure 4. Pressure-Density Isotherms for Composition A Made with 90/10 Petrocene C - Alox 600; Ten-gram, 1" Pellets. Dotted Curves Represent Densities of Re-pressed pellets.

operator to hold the sample at reduced pressure (15 to 18 mm.) for 5-10 minutes before applying the pressure. Composition A's pressed in the vacuum mold showed no re-expansion effects; hence it was decided that systematic studies should begin with vacuum pressing, that the pressing at atmospheric pressure should follow, and that then the re-expansion phenomenon could be isolated and studied by itself.

(3) Systematic Pressing of Pellets

a Vacuum Pellets

In order to cover the range of penetrations between 15 and 150, Composition A's were made up from four different waxes, Superla Wax Yellow, Bruceton Wax No. 10, Yellow African Beeswax and 95/5 Gulf Petrowax A-100 600, with the aid of which this entire range could be covered within the temperature range of 25 to 40°. Pellets were pressed at a series of pressures and penetrations, the former being restricted to the range between 2000 and 10,000 pounds per square inch. Figure 5 shows the results of these pressing experiments presented as penetration-density isobars. An alternative method of presenting these results is to plot pressure-density curves at constant penetration,

is shown in Figure 6. There is a tendency for the curves to come very close together at high pressures and penetrations, so that it has been necessary to change the spacing of the curves from 5 units to 10, and finally to 50. This can be eliminated, and the equivalent roles of the pressure and the penetration can best be shown by drawing lines of constant density as in Figure 7. This graph makes very clear the fact that there are many combinations of pressure and penetration which will lead to the same resulting density. At the lower pressures and penetrations the curves are particularly simple, being practically linear. At the higher pressures and penetrations, a limiting density is apparently being approached; 1.636 is the highest value that has been obtained in this vacuum mold, and it seems doubtful if values above 1.640 would be obtained without going to very much higher pressures, even though the theoretical value of the limiting density is 1.683, assuming that no wax is lost in pressing.

b Ordinary Pellets

Pellets were next pressed under conditions which duplicated as closely as possible those of the preceding section, except that the explosive was not evacuated before applying the pressure. The resulting densities are shown as a function of the penetration in Figure 8, where the solid curves are the curves for vacuum pressing taken from Figure 5 for comparison. The irregularity of the results is such that it was deemed best not to attempt to draw curves through the points; the dotted lines are merely intended to connect the points for a given pressure, to avoid confusing them. The serious

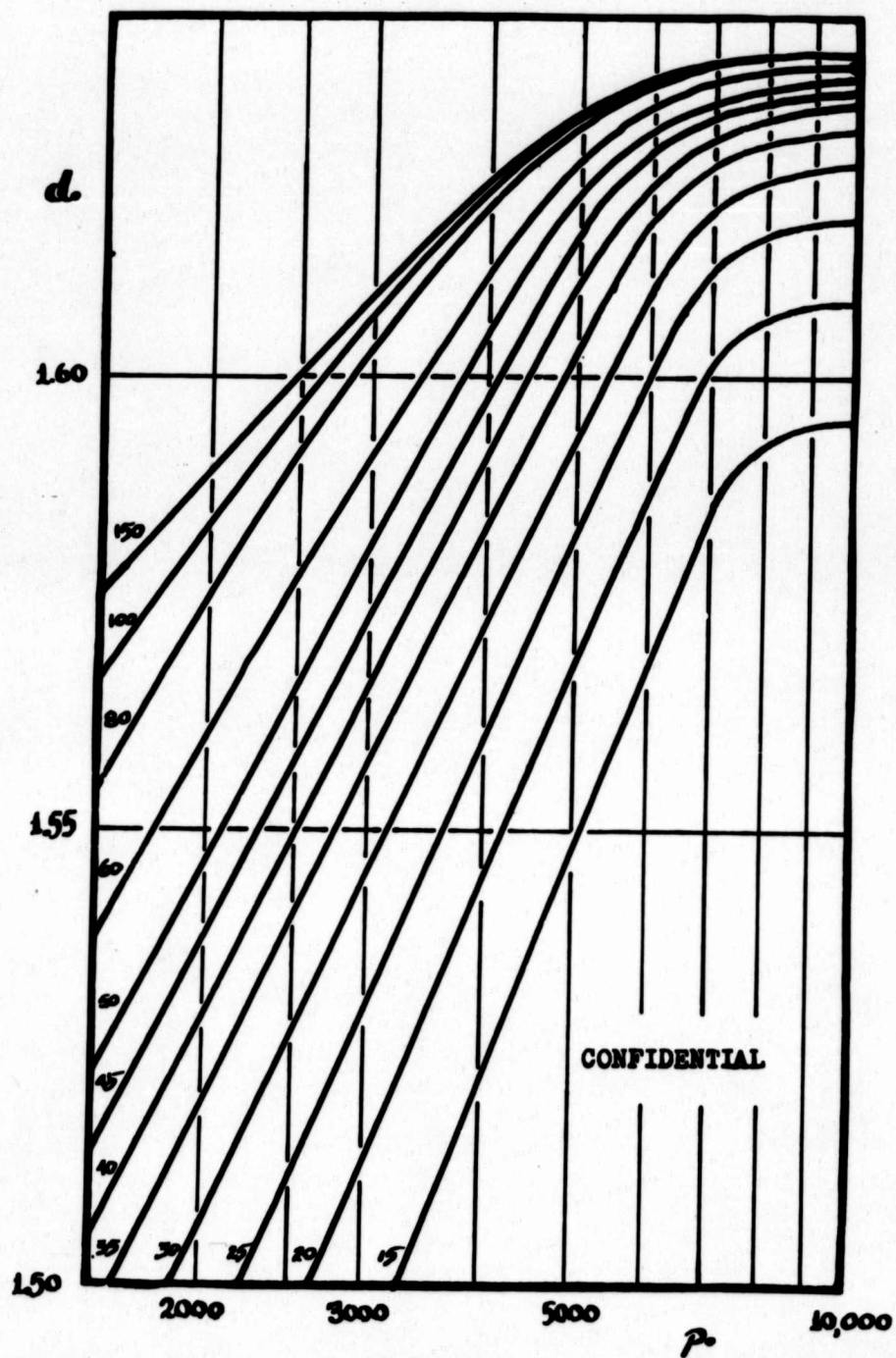


Figure 6. Pressure-Density Curves at Constant Penetration; Ten-gram, 1", Vacuum-Pressed Pellets.

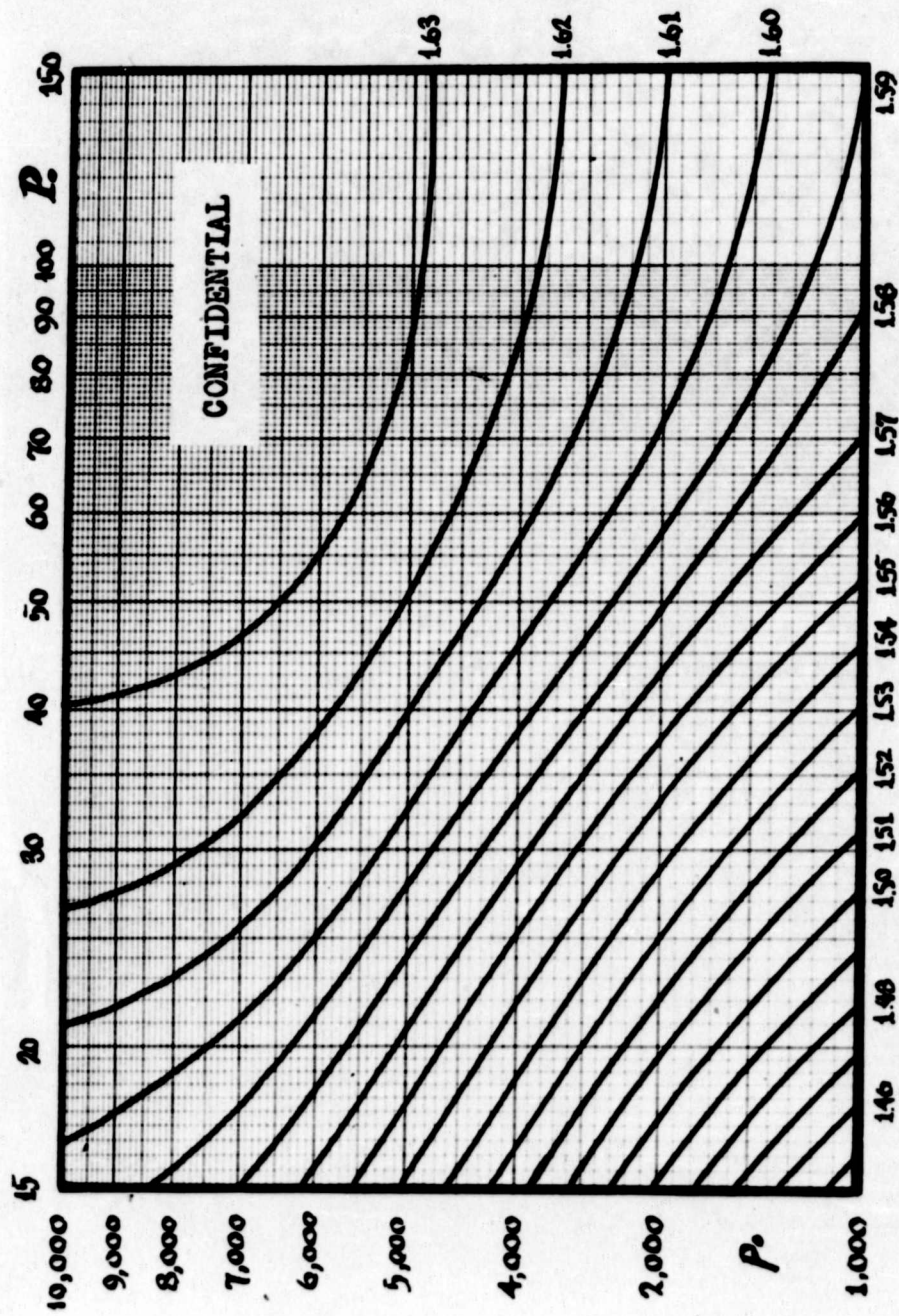


Figure 7. Pressure-Penetration Curves at Constant Density;
Ten-gram, One-inch, Vacuum-Pressed Pellets.

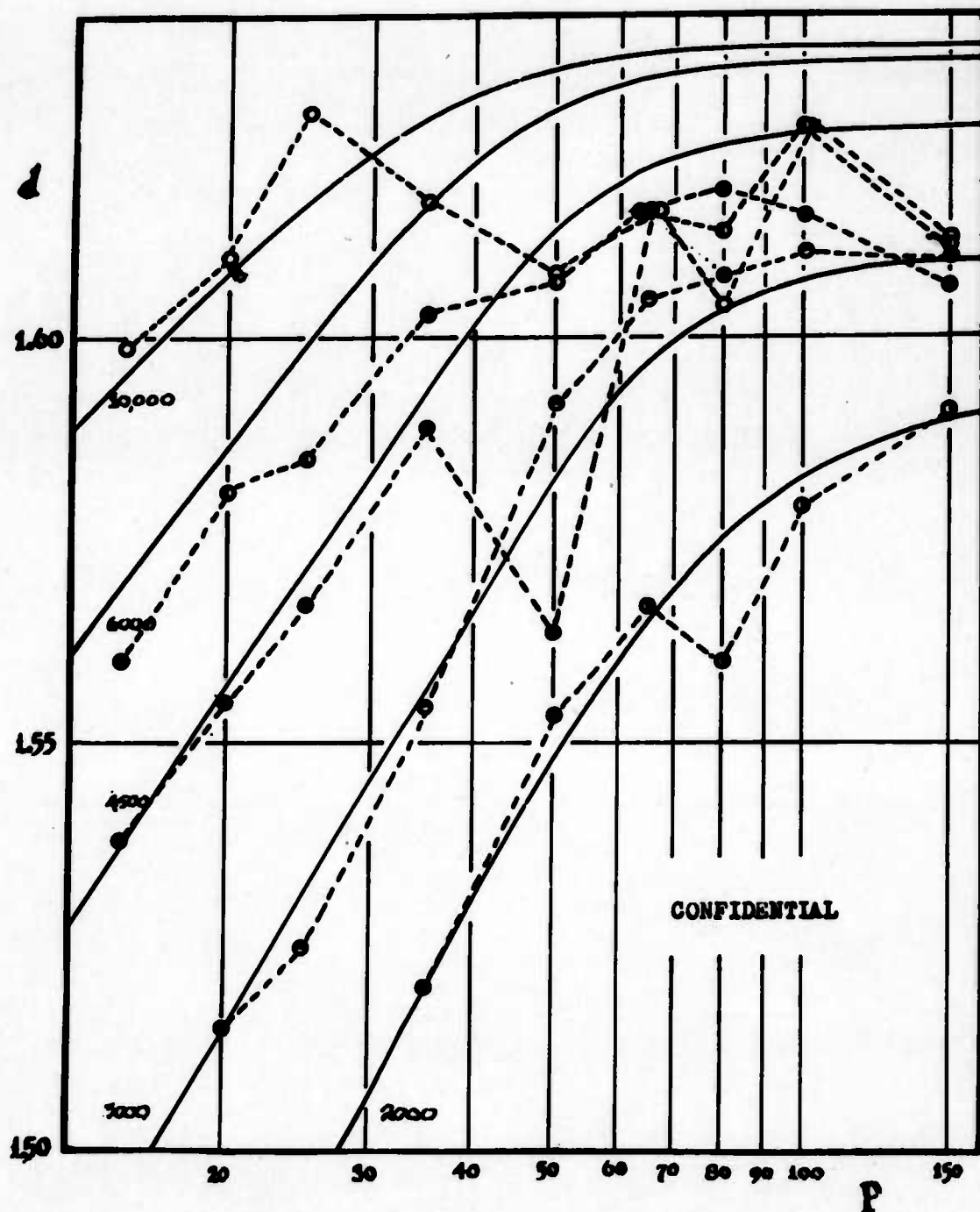


Figure 8. Penetration-Density Isobars for Pellets Pressed at Atmospheric Pressures; Solid Curves from Figures 5.

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re-expansion is seen to be limited approximately to the region bounded by $d = 1.60$ and $P = 30$.

It would appear that the re-expansion effects are as great under these conditions as they will ever be, because the walls of this mold are very smooth, offering little resistance to re-expansion; the plunger fits very snugly, offering very small opportunity for air to escape; and the Composition A itself has very porous grains, which probably contain much more entrapped air than the grains of harder material like the British product.

These results also show that for the higher pressures, increasing the penetration (by raising the temperature, for example) will lead to decreases in density, if the initial value of the penetration is around 80 to 90 (see the solid curves of Figure 4), but will lead to increases in density if the initial penetration is well below these values (such as is the case in Figures 2 and 3). It is also clear from these results that the irregular parts of the 50° curves of Figures 2 and 3 should be confined to their high-pressure parts.

It was hoped that the re-expansion might be expressible as a correction factor, which would be a function of the pressure and penetration, and had simply to be added to the results of the vacuum pellet curves. In view of the irregularity of the densities themselves, when re-expansion occurs, it seems unwise to attempt to give quantitative treatment to this re-expansion correction. It will be clear from Figure 8 in what regions of pressure and penetration one may expect the effect to be appreciable, and the approximate extent of the re-expansion may also be estimated.

c The Effect of Pellet Diameter

To check the effect of pellet diameter upon the density of pressed pellets, some two and three-inch diameter pellets have been pressed. The following results show that the density is apparently independent of the diameter, but depends strongly on pellet length; pressure employed was 3000 psi.

| Diameter | Length | B.Wax No. 10 20°C. | B.Wax No. 10 30°C. | Petroceno-C-Alox 20°C. |
|----------|--------|-----------------------|-----------------------|---------------------------|
| 2" | 2" | 1.495 | --- | 1.559 |
| 2" | 1" | 1.522 | --- | 1.591 |
| 1" | 1" | 1.538 | --- | 1.598 |
| 1" | 1/2" | 1.542 | --- | 1.608 |
| 1" | 2" | 1.499 | 1.534 | 1.560 |
| 3" | 2" | --- | 1.547 | --- |

It is hard to be certain that various diameter molds are of equal

clearance (see section e. following), but we feel that within diameter variations from one to three inches there is shown no clear effect of diameter on density. The effect of pellet length, on the other hand, is unmistakable.

d The Effect of Pellet Length

Pellets have been pressed in the one-inch mold at various pressures and penetrations, in lengths varying between 1/8" and about 2"; it is somewhat more convenient in practice to vary the weight of explosive pressed, than to vary the length of the pellet. The results for one-inch pellets may be expressed as follows:

- (a) For pellets smaller than 10 g., the density begins to decrease with decreasing pellet weight; this fact is of no special practical interest, because such thin pellets would surely never be pressed in practice.
- (b) The overall density for larger pellets may be considered as essentially those given in Figure 6 for pellets between 10 and 15 grams.
- (c) Approximate values for the overall pellet densities of pellets larger than 15 g. may also be determined from the curves of Figure 6, by using instead of the actual press pressure an effective pressure p' , given for a one-inch pellet of mass m by:

$$p' = p_0 - 0.0135 (m - 15)$$

Because of the diameter independence of pellet density, the above statements also hold for any cylindrical mold of diameter D , inches, if the values of m are replaced by m/D^2 in the equation, and $10D^2$ and $15D^2$ g. replace 10 and 15 g. in the sections (a) and (b) above.

It should be noted that these simple relations hold only in the absence of re-expansion, and the calculated densities are to be considered as upper limits under conditions where this phenomenon is to be expected.

The approximate "effective pressure" formula of section (c) above implies that when pellets are longer than about 3/4", the average effective pressure begins to decline with increasing length. When working with gage pressure--penetration combinations well above the knee of the pressure-density curves, this decline in effective pressure will not be reflected so readily in density decline as when one is working below the knee.

o The Effect of Plunger Clearance

When pellets are pressed in molds other than the one with which the data of Figures 5, 6, 7 and 8 were obtained, a tendency to give densities higher than those predicted from these curves was noted. It is felt that this is the result of the fact that the plunger of the vacuum mold was more tightly fitted than the plungers of molds not designed for evacuation. The data with a given mold may be brought into agreement with the data of the above-mentioned curves if the pressing pressures are multiplied by a constant factor, characteristic of the mold; this factor may be readily determined by pressing a few pellets, under conditions of known penetration, in the mold.

f The Effect of Time of Pressing

There has been no systematic study of pellets pressed for differing lengths of time; all the work cited employed one minute application of the pressure. It is known that 10 g. pellets pressed for 10-sec. have somewhat lower densities than the one-minute pellets, and that times in excess of one minute will increase the density of pellets, especially the longer pellets. It is believed that the results for times other than one minute would probably be brought into correlation with the one-minute data by finding a multiplicative factor for each time (unity for one min.) which would relate the press pressure and the effective pressure to be used in connection with curves such as those of Figure 6.

g The Effect of Grain Size of Explosive

Experiments were made on the pressing of British Composition A of varying grain-sizes, at 20° and 6000 lb/in². Unmilled particles of the material, of size about 3/16"D, and also material crushed in the mill and screened were used. The results were:

| | |
|---------------------------------|-------|
| Unmilled | 1.622 |
| On Tyler #10 | 1.623 |
| On Tyler #35, through #20 | 1.618 |
| On U.S. #100, through Tyler #35 | 1.619 |
| Through U.S. #100 | 1.605 |

The only sample which shows a significantly large drop in density is the fine sample, and this may have contained uncoated RDX.

h The Effect of Consistency of the Explosive Grains

As mentioned previously, the grains of British Composition A are more compact and harder than the grains of material prepared in our laboratory. The only result of this difference that we have been able to observe surely is that the densities

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decrease more rapidly for low pressures with the hard material than with the soft, but even this effect is small under conditions of pressure and temperature which yield densities above 1.50, so its practical importance is negligible. It was thought that the harder and more compact grains might cause less of the re-expansion trouble than the softer, more open grains, but there seems to be actually no concrete evidence for this belief.

i The Variation of Density Within a Pellet

Up to this point we have concerned ourselves completely with the overall densities of pellets; let us now consider the variation of density along the length of cylindrical pellets. The consistency of the pressed material is such that pellets cannot be neatly sectioned, so charges were made up in ten-gram increments, hand-pressed into the mold with a circular tin-foil separating diaphragm between each increment. The entire charge was then pressed, and when removed from the mold, could be separated into ten-gram sections. These sections did not have plane faces, except where they were in contact with the plungers, so their volumes were determined by placing them in a container of known volume, and filling the container with methanol from a buret. The pellets were lightly coated with vaseline to avoid absorption of the liquid. The densities of such sections, as a function of their average distance, in cm., from the moving plunger, are shown in Figure 9, for various pressure-penetration combinations. It is interesting to observe that for higher densities pressure-penetration combinations giving the same overall density give also the same dependance of density upon length. Since these combinations lie in the linear portions of the curves of Figure 6, and since the curves of Figure 9 are also essentially linear, the results mean that the effective pressure within the mold is an exponentially decreasing function of the distance from the moving plunger, in the first approximation.

To determine radial variations in density of pressed Composition A pellets, 2" diameter 1" high pellets of Composition A made with Bruceton Wax No. 10 were pressed to high density, and holes bored in them. The results follow:

| Av. Density of Original Pellet | Size of Hole Bored | Av. Density of Bored Pellet | Change in Av. Density |
|-----------------------------------|-----------------------|--------------------------------|--------------------------|
| 1.6105 | 1/2" | 1.6088 | -.0017 |
| 1.6109 | 3/4" | 1.6079 | -.0030 |
| 1.6189 | 1" | 1.6144 | -.0045 |

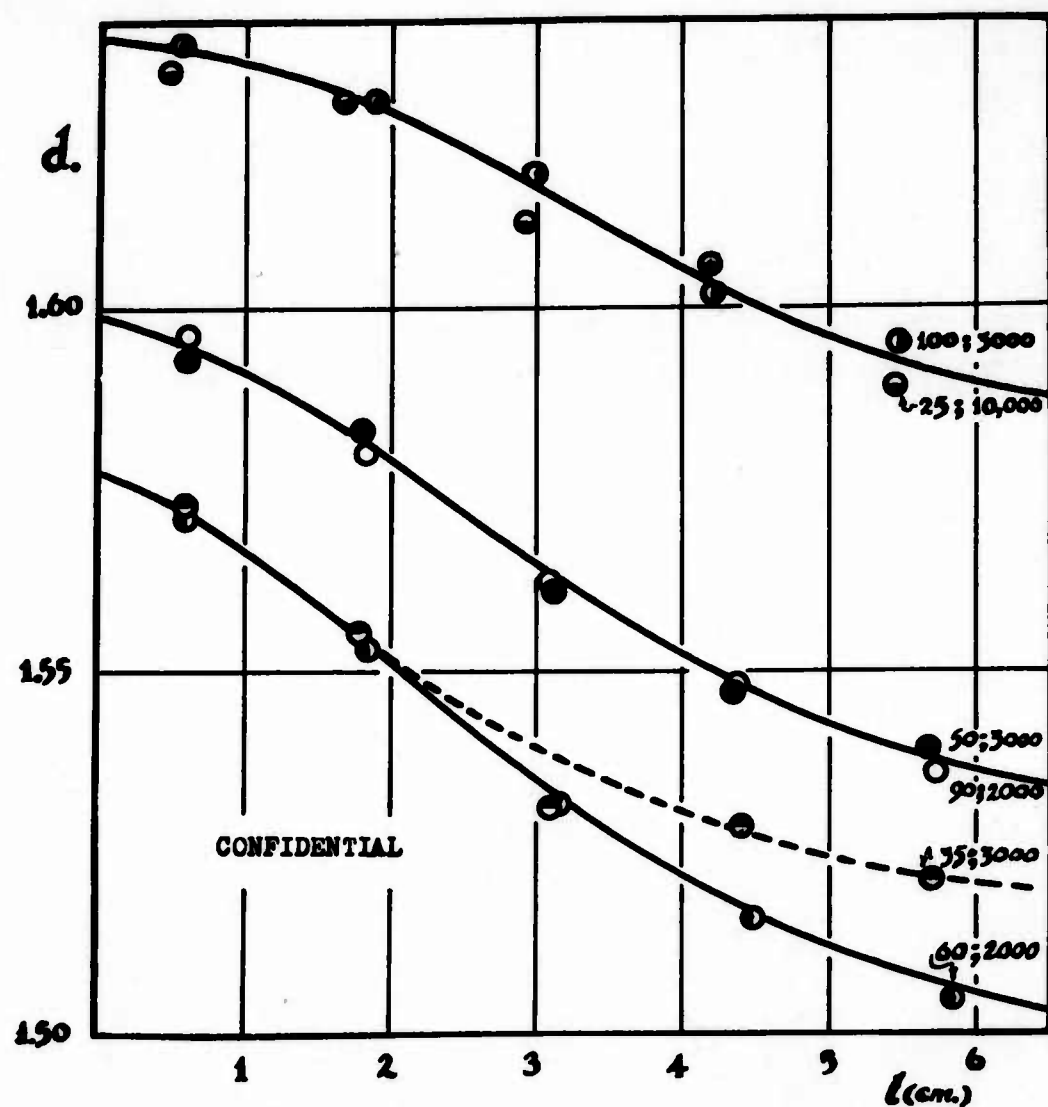


Figure 9. Average Density of Pellet Sections as a Function of Mean Distance from Moving Plunger.

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In pressing large charges of Composition A, some difficulty might be encountered in the effect of the way the powder is spread out in the mold before pressing. To determine the effect of non-uniform distribution of Composition A before pressing, a 2" pellet was pressed, with the mold lying on its side. The weighed amount of Composition A made with Bruceton Wax No. 10 was put into the 2" mold, and the plunger put in until it just touched the surface of the powder. Knowing the length of mold and plunger, the volume of unpressed powder was determined. The plunger was then pulled up far enough out of the mold to give an equal free volume. The mold was then turned on its side so that the powder could flow down, and concentrate on one side of the mold. Two pellets were pressed this way. The bottom side of the pellet was marked after pressing. The overall densities of the pellets were measured, and then the pellets were cut in half, separating the top and bottom sections. Both sections were sanded down, measured geometrically and their densities determined, with the following results:

| | 5,000 psi. | 10,000 psi. |
|------------------------|------------|-------------|
| Overall density | 1.5570 | 1.6107 |
| Density of top half | 1.5539 | 1.5988 |
| Density of bottom half | 1.5525 | 1.6092 |

In neither case does there appear to be a significant density difference between the two halves, though the irregularity in powder distribution was great.

(A) Shell Loading with Composition A

a Loading Method

The base of a 37 mm. M63 shell was provided with an adapter to hold a 0.882"D plunger; a support for the nose to hold the assembly upright in the vertical press, and an electrically heated water bath to control the temperature completed the loading equipment. Explosive was loaded into the shell-adapter assembly, the unit was brought slowly up

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to the desired temperature, held there for 15 minutes, and then pressed for one minute. The explosive was loaded in two and three increments, and at various pressures and temperatures.

The weight of the increment was determined by weighing the shell before and after the addition; the volume was measured by determining the free volume with methanol added from a buret, and subtracting this from the total volume of the cavity. Shell fillings were also pressed in which the last increment was pressed with a plunger tipped with a booster-cavity former; the booster cavities formed in this way were strikingly sharp and clean, and the use of such a tip for the last increment has no bad effect on the overall density.

Unfortunately, the sizes of the increments were not always kept quite constant, so that it is not easy to discuss the densities of the various increments separately. As would be expected, the densities of the increments pressed in the ogival portion of the cavity are abnormally low, and the densities of the increments pressed in the cylindrical portion of the cavity are about normal. There seems to be less tendency for the filler to re-expand in the shells than in the smooth cylinder molds, and above the melting-point of the wax its tendency to exude during pressing seems also to be less.

b Interpretation of Results

The overall density of the filling of a shell may be predicted at any press pressure and penetration, for any given number and weight of increments, if the overall density is known for this pressing schedule at one pressure and penetration, or, better, if it is known for a few combinations of pressure and penetration. This correlation again employs Figure 6, and is certainly approximately correct. A shell is pressed at a definite press pressure p and a definite penetration P , according to a fixed schedule--number and weight of increments, time, etc.--yielding an overall density d . Reference is then made to Figure 6, and the pressure p' corresponding to the density d and the penetration P is determined. The ratio p'/p , which will in

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general be less than unity is a constant characteristic of the pressing schedule and the shape of the shell cavity. The value of this constant for the 37 mm. shell, loaded with a first increment of 29 g., pressed one minute with a flat plunger and a second increment of 20 g., pressed for one minute with the tipped plunger, is 0.580. For the same shell loaded with a first increment of 16-1/2 g., a second increment of the same size, each pressed for one minute with a flat plunger, and a third increment of 17 g. pressed for one minute with the tipped plunger, the value of the constant is 0.760. The value of this constant could be further increased by increasing the number of increments, or by increasing the time of pressing each increment, but it can probably not be made as great as unity.

Unfortunately, much of the shell pressing was done early in the course of this study, at temperatures outside the range in which the penetrations have been measured; accordingly, there is not abundant evidence to support the statements of the above paragraph in a striking fashion. The curves of Figure 10 represent the overall densities for two-increment pressings at various temperatures and pressures. Beside the scale of p , the scale of p' is also shown, and the solid curves joining the experimental points are members of the family of Figure 6, on the p' scale.

Some shell pressing has also been done at 10 sec., instead of our regular time of one minute, and lower densities have been found; a slight increase in the pressure would, however, be enough to bring them up to the one-minute values.

(5) Effects of Rolling on Pressing Characteristics

These studies were all made with samples of Composition A, and no detailed comparisons of the pressing properties of Composition A and Composition A-3 have been made. A good many samples of the latter have been pressed from time to time, and it is our belief that although slightly lower densities are obtained with unrolled products, the general relations are not much altered so long as the RDX is well coated. Poorly coated unrolled products, however, give abnormally low densities and very weak pellets.

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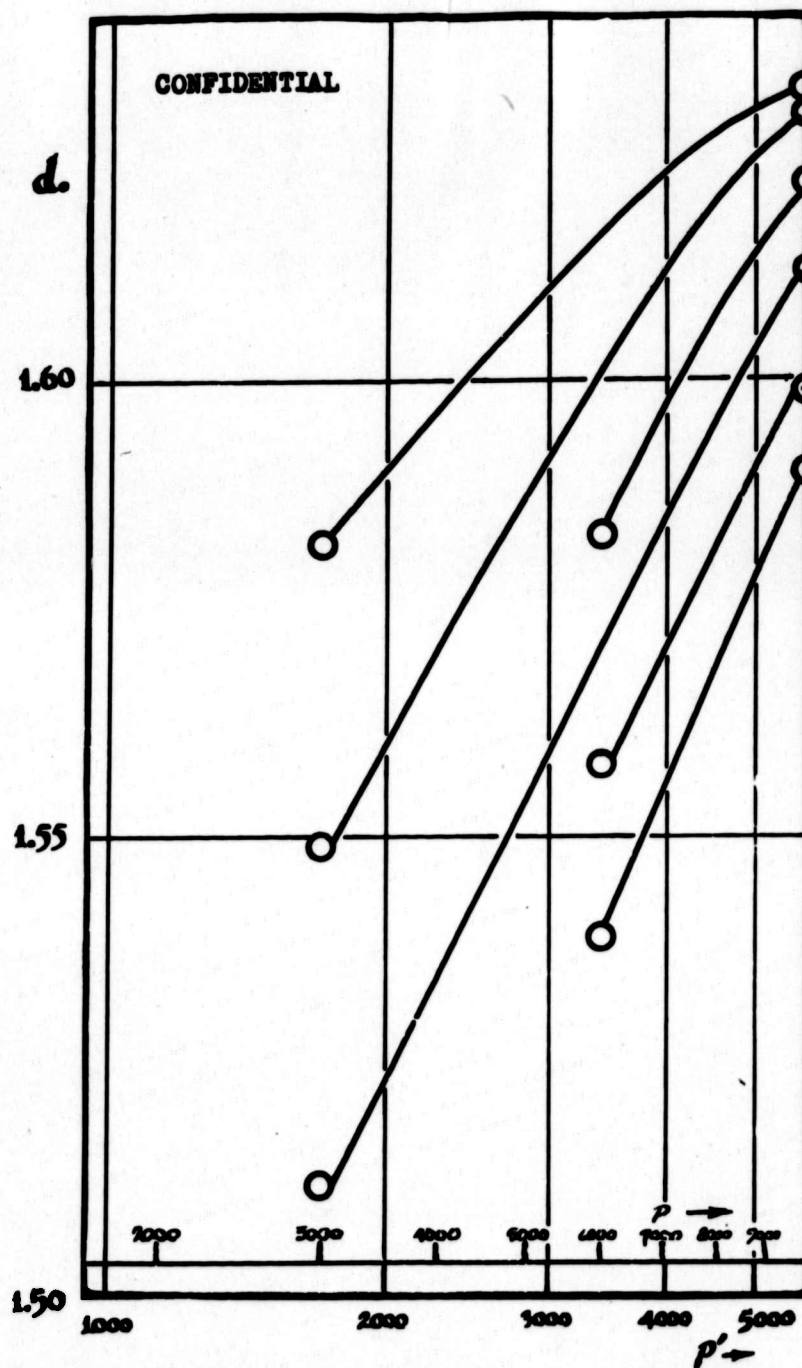


Figure 10. Overall Density of Shell-Load as a Function of Press Pressure (p), and Effective Pressure (p'), for Two-Increment Pressing at One Minute Hold.

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(6) Stickiness of Composition A

It has been noticed both in Navy Shell loading and also in the laboratory (particularly with the Stokes pellet-press) that, especially during warm weather, there is a tendency for Composition A to stick to dies, mold-walls, and booster-formers. This tendency is related to the stickiness and to the softness of the wax. No thorough study has been made of this matter, but it suggests the value of choosing for Composition A waxes which are not sticky, and which have rather low penetrations. Alox-600 increases the penetration and stickiness of waxes; accordingly, it seems wise to reduce the Alox content, which was chosen originally as 10% in a completely arbitrary manner, to the lowest level which gives suitable coating properties; one percent seems adequate. The wet coating of Composition A with graphite might also reduce stickiness.

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4. Explosive Properties of Composition A

(1) Sensitivity

a. Effects of the Properties of the Wax on Sensitivity

Early investigations of waxes as substitutes for beeswax in Composition A, in which the relatively irreproducible Type 4 Impact Machine was mainly employed as the method for assessing degree of desensitization, created the impression that of a series of petroleum-base waxes of similar physical properties, some were, while some were not good desensitizers. It is probably not unfair to state that the ten waxes recommended in OSRD Report No. 313 were chosen from many studied in large part because they were believed to be particularly effective desensitizers. We now largely reject this concept of specifically high desensitizing ability, unrelated to gross physical properties, and feel that any wax having a penetration suited to pressing operations, proper stability characteristics when mixed with RDX, and the ability to coat well from water will also be a suitable desensitizer. Subtle differences in desensitizing action may yet exist among waxes of similar apparent physical properties; such differences are precisely the ones which are very difficult to assess with certainty^{9/}. At least it seems unwise to conclude that a certain wax is highly superior to other similar ones unless this can be demonstrated by a number of different sensitivity tests - in other words, the basic assumption that waxes are alike in this respect seems to us more reasonable and more fruitful than the opposite assumption that their desensitizing abilities may vary capriciously. Examples of impact and bullet-test results of Composition A samples made with various waxes of suitable penetration and under circumstances in which good covering of the RDX was achieved are given below:

^{9/} See Picatinny Arsenal Technical Report No. 1489, for relative desensitizing action of two waxes on PETN.

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Drop-Weight Impact Tests-Composition A

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| <u>Wax</u> | <u>50% Pt(cm); Type 4</u> | <u>Index; Type 6</u> |
|-----------------------------|---------------------------|----------------------|
| Beeswax | 0% at 100 | 87 |
| Bruceton Wax No. 10 | 28% at 100 | 87 |
| Petrocene C - Alox 600 | 0% at 100 | 121 |
| GPD-702-C* - Alox 600 | 94 | 90 |
| Gulf Petrowax B - Alox 600 | 0% at 100 | 109 |
| Gulf Petrowax A - Alox 600 | 20% at 100 | 107 |
| Cerese Wax Brown - Alox 600 | 98 | 115 |
| Product 2300* | 33% at 100 | 107 |
| Product 3000** | 5% at 100 | 94 |
| TNT (control) | 0% at 100 | 102 |

* Socony-Vacuum Oil Co.

** Moore and Munger

Drop-Weight Impact Tests-Composition A-3

| <u>Wax</u> | <u>50% Pt(cm); Type 12</u> |
|---------------------------------|----------------------------|
| Beeswax | 75 |
| Bruceton Wax No. 10 | 75 |
| Petrowax A - Alox 600 | 69 |
| Beeswax (British Rolled Sample) | 70 |

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OSRD No. 5744

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Bullet Tests - Comp. A

Loaded at density 1.0 in 1" p.n. Cal. 0.30 Ball

| <u>Wax</u> | <u>No. Trials</u> | <u>Result</u> |
|---------------------------------------|-------------------|---------------------------|
| Beeswax | 5 | 5 inert |
| Bruceton Wax No. 10 | 14 | 14 inert |
| Victory Wax - Alox 600 | 10 | 10 inert |
| Petrocene C - Alox 600 | 5 | 5 inert |
| Petrowax A - Alox 600 | 4 | 4 inert |
| Barnsdall Special Wax, Amber-Alox 600 | 4 | 4 inert |
| Esso Decoiled Petrolatum - Alox 600 | 4 | 4 inert |
| Paraffin - Alox 600 | 4 | 4 inert |
| Petrocene C - Lanolin | 5 | 5 inert |
| Petrowax A - Lanolin | 5 | 5 inert |
| Standard Refined Wax - Alox 600 | 10 | 10 inert |
| Stanolind 160/65 - Alox 600 | 10 | 10 inert |
| TNT (control) | 15 | 10 inert 5 detonations |

Test results of this sort have caused us tentatively to accept the notion that if a wax is otherwise suitable for Composition A, its desensitizing action will be adequate.

b. Comparison with other Explosives

Composition A is rather unlike such materials as TNT and Ammonium Picrate, both in physical properties and also in being composed of a large amount of sensitive material plus a relatively small amount of desensitizer. These differences make it hard to obtain unique sensitivity orderings for Composition A and such other explosives under varied conditions of impact.

(a) Drop-Weight Impact Tests

There are now available, in the preliminary results of the U.S. Navy Comparison Series of Impact Tests, test-results for a uniform sample of Composition A-3 tested by several laboratories on machines of their own designs. These results, and several others from ERL machines are presented in the following table; for each machine the relative 50% Points (TNT = 100) are given:

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| Explosive | Machine | | | | ERL | ERL | ERL | Average |
|------------------|-------------------------|----------|------------------|--------|------------|-----------|-----------|------------|
| | duPont, N.P.F.* Cup. | Hercules | N.R.C. Type 5 | Canada | Type 12 | Type 7 | Type 9 | |
| Ammonium Picrate | 98 | 47 | 65 | 100 | 144 | 187 | 147 | <u>113</u> |
| Comp. A | 86 | 83 | 54 | 293 | 44 | 97 | 91 | <u>107</u> |
| TNT | 100 | 100 | 100 | 100 | 100 | 100 | 100 | <u>100</u> |
| Tetryl | 65 | 28 | 42 | 37 | 22 | 62 | 63 | <u>46</u> |

*Naval Powder Factory, Indian Head, Md.

These average values will not be greatly changed by omitting any one result, except that of the NRC, Canada, which gives so high a value for Composition A. Omitting this set, the order becomes:

| | | |
|------------------|---|------------|
| Ammonium Picrate | - | <u>115</u> |
| TNT | - | <u>100</u> |
| Composition A | - | <u>75</u> |
| Tetryl | - | <u>46</u> |

It seems fair to conclude generally from these tests that Composition A is on the average more sensitive than Ammonium Picrate and less sensitive than Tetryl. Under most conditions it seems somewhat more sensitive than TNT, but at least under the condition of impact obtaining in the Canadian Machine it may behave less sensitively than TNT.

(b) Bullet Impact Tests

Tested at density 1.0 in 1" pipe-nipples, with Cal. 0.30, Ball, M2 ammunition, Composition A compares with TNT and Ammonium Picrate in the following way:

| <u>Explosive</u> | <u>No. Trials</u> | <u>% Inert</u> | <u>% High Order Det.</u> |
|------------------|-------------------|----------------|--------------------------|
| Ammonium Picrate | 11 | 100 | 0 |
| Composition A | 59 | 100 | 0 |
| TNT | 15 | 67 | 33 |

Under these circumstances also, as in the NRC, Canada, drop-weight test, Composition A is less sensitive than TNT. The test does not make any differentiation between Composition A and Ammonium Picrate.

(c) Booster Sensitivity Test

The booster sensitivity test^{11/} gives the following results and order of increasing sensitivity for pressed charges at density 1.6,

| <u>Explosive</u> | <u>Limiting Boosters*</u> |
|------------------|---------------------------|
| Ammonium Picrate | 5//20+2 |
| Composition A | 20+2//15+2 |
| TNT | 15+2//10+2 |
| Tetryl | 5+2//20+3 |

*The notation 5//20+2 means, for example, that the explosive detonated with a 5 g. Tetryl booster, but failed with a 20 g. Tetryl booster separated from the charge by 2 cm. of Aerawax B.

On the other hand, TNT pellets of density 1.6 were not detonated by a duPont No. 8 Tetryl-base detonator set with base flush with the pellet surface, while under similar circumstances Composition A pellets often detonated. It is felt that Composition A, with its highly sensitive RDX crystals, might well be more sensitive than TNT to the attack of this detonator, which is a small shaped charge, whereas it is less sensitive to the attack of the members of the graded booster series, which throw no small, fast metal particles.

(d) Large Scale Results

Plate-tests of Composition A-3 loaded A.P. shell have been made by the U.S. Navy. The details of these tests are not known to us, but it has been reported that whereas the 3" shell, so loaded, passed the plate-tests, the 6" shell failed. These results show that Composition A is more sensitive than Ammonium Picrate under these conditions. Mass detonation (sympathetic detonation) trials with the Composition A-3 loaded 5"-38 A.A. shell show that such shell detonate en masse under circumstances under which the same shell loaded with Ammonium Picrate do not. This result is to be related not only to the apparently higher booster-sensitivity of Composition A-3, but also to the higher performance of the explosive, which provides much greater impulse to mass detonation. Comparisons with TNT are hard to make, because it is usually cast-loaded in such shell, and in order to draw conclusions from full-scale trials which should be expected to correlate with small-scale ones, one must make allowances for this difference in loading; how in detail to make such allowances is not now known.

^{11/}

See OSRD No. 5746 for details.

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(e) Conclusions

The various tests which have been cited make it fairly clear that in general Composition A is a definitely more sensitive explosive than Ammonium Picrate, but is definitely more insensitive than Tetryl. It differs basically from pure-compound explosives, however, as a consequence of which (at least in part) it displays under certain conditions greater, and under other conditions less sensitivity than TNT.

(2) Performance

Although Composition A is in roughly the same sensitivity class as TNT, and greatly less sensitive than Tetryl, it outperforms them both in all small-scale tests we have made. The following are representative values of our laboratory measure of performance^{12/}.

| Explosive | Ballistic Mortar (T.V.) | Relative Brisance dens. (T.V.) | Relative Fragment Vel. dens. (T.V.) | Velocity of Detonation dens. V(m/sec) |
|------------------|-------------------------------|--------------------------------------|---|---|
| Ammonium Picrate | 99 | -- * | -- -- | 1.55 7050 |
| TNT | 100 | 1.602 100 | 1.580 100 | 1.60 6850 |
| Composition A | 135 | 1.611 126 | 1.608 108 | 1.60 8200 |
| Tetryl | 130 | 1.592 115 | -- -- | 1.55 7375 |

*This value is not known, but the value 100 has been obtained for Picratol, 52/48; hence the value for Ammonium Picrate at high density must be near 100 also.

Brisance as measured by the plate-denting test, the fragment velocities, and the rate of detonation all depend upon density. In the previous table we cite the densities at which these data were determined. With respect to TNT, the two explosives Ammonium Picrate and Composition A (each suitable only for press-loading) occupy complementary, not similar, positions. Ammonium Picrate is similar to TNT in performance, but exceeds it in insensitivity; Composition A is similar to TNT in sensitivity, but exceeds it in performance. Composition A displays both higher performance and greater insensitivity than Tetryl.

^{12/}

See OSRD Report No. 5746 for further details of these tests.

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5. Stability of Composition A

(1) Chemical Stability

All seriously-considered experimental Composition A samples have been subjected to routine 135°C Thermal Stability and 120°C Vacuum Stability Tests. No samples have been acid within 300 minutes in the former. All samples made with petroleum-base waxes have shown very satisfactory Vacuum Stability at 120°C; as remarked in OSRD Report No. 313, p. 11, most samples display a Vacuum Stability about that of pure RDX. Synthetic waxes do not, however, have the chemical uniformity of petroleum waxes; some do and some do not form as stable mixtures with RDX as the petroleum waxes. Typical results are the following:

Vacuum Stability Tests; Composition A

| <u>Wax</u> | c.c. gas evolved at 120°C | |
|--|---------------------------|-----------------|
| | <u>90 minutes</u> | <u>48 hours</u> |
| <u>Petroleum Waxes</u> | | |
| Victory Wax | 0.28 | 0.48 |
| | 0.52 | 0.30 |
| Stanolind 170/75-Alox (10%) | 0.78 | 0.87 |
| | 0.51 | 0.58 |
| Black Aristowax-Alox (10%) | 0.32 | 0.70 |
| | 0.54 | 0.73 |
| Aristowax-Alox (Br. Wax No. 10) | 0.09 | 1.05 |
| | 0.14 | 1.03 |
| Aristowax-Lanolin (10%) | 0.48 | 0.35 |
| | 0.45 | 0.67 |
| <u>Synthetic Waxes</u> | | |
| Glyco Wax A - Alox (1%) | 0.57 | 0.70 |
| | 0.70 | 0.80 |
| [Glyco Wax A + 5% Stearamide]-Alox (10%) | 0.61 | 0.74 |
| | 0.65 | 0.38 |
| Acrawax B - Alox (1%)* | 0.85 | 9.40 |
| | 0.93 | over 12 |
| Carbowax 6000 | 4.18 | over 12 |
| | 4.17 | < 24 hours |
| | | <u>at 100°C</u> |
| Carbowax 6000 | 2.28 | 8.35 |
| | 2.30 | over 12 |

*It has already been pointed out in Picatinny Arsenal Technical Report No. 1487 (Jan. 1945) that the Acra waxes are incompatible with RDX.

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(2) Corrosion

Corrosion tests (compatibility tests with brass and steel in a humid atmosphere) of Composition A were reported in OSRD Report No. 397. No others have been made. It has been observed that stearic acid, as an additive to waxes, reacts with brass and colors the explosive green. Beeswax does this also without additives.

(3) Exudation

One of the more difficult properties of an RDX-wax mixture to measure and interpret is its behavior on hot storage, its so-called exudation. The numerical values obtained for percent of wax lost after varying periods of storage at any given temperature have only relative significance, being used merely to determine the relative merit of the waxes tested. It is not known that even waxes which appear, by such comparison, to be very poor in the laboratory will not be satisfactory when loaded into an actual munition, since no full scale test has ever been run. It is believed, however, that loss of low melting wax or oil at elevated temperatures might have serious results. While it is doubtful whether any of the wax can leak out of the loaded shell, it is possible, since some 5% voids exist even in a fairly good pressed load, that the wax or some part of the wax may flow away from some of the RDX leaving one or more areas in the mass of explosive with less than the amount of wax necessary for adequate desensitization. Such high RDX-low wax areas would be more sensitive, and might cause premature detonation of the shell.

Aside from considerations of effectiveness in desensitizing, the selection, in 1942, of Beeswax substitutes from the many available petroleum waxes was based mainly on the exudation results (OSRD Report No. 397). Since that time, the object of such exudation investigations at this laboratory have been to find the best possible wax from the standpoint of exudation, rather than one which will merely match Beeswax. All of the original exudation tests as described in OSRD Report 397 were done at 50°C with 5 g samples enclosed in glass weighing bottles. Since that time, it has been found convenient to use 10 g sample, 1" in diameter, and since no difference was observed whether the sample was enclosed in a weighing bottle or not, all samples were stored in

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the open. Moreover, experience in tropical storage of munitions has led to interest in behavior at higher temperatures; 60°C has been suggested as a standard test-temperature, and is now so used by us.

a. Procedure

10 g of Composition A are pressed in a 1" mold at 10,000 psi. The end of the resulting pellet which was next to the moving plunger is sanded until it is perfectly flat. The pellet is weighed, and then with the smoothed surface down, is placed on 10 pieces of filter paper 1-1/2" in diameter. A 75 g brass weight, 1" in diameter, is placed on top to insure good contact between the pellet and paper. The whole assembly is then placed on a wire screen and stored in a forced-draft oven at 60°C. The pellet is removed and weighed after 3 days, 1 week, 2 weeks, 5 weeks and 12 weeks. The results are expressed as percent of wax lost.

b. Results

Recent exudation studies have revealed two very important facts: (a) decreasing the Alox content from 10% to 1% cuts the exudation; (b) the Aristowax 160/65 now being produced is considerably inferior, from the standpoint of exudation, to the Aristowax 160/65 recommended by this laboratory in 1942; other waxes exist which are superior in this respect to current production Aristowax.

The following Table demonstrates, by some typical examples, the effect upon exudation of lowering the content of Alox 600.

| Wax | Percent Wax Lost; 2 weeks at 60°C. | |
|-------------------------------|---------------------------------------|----------------|
| | <u>10% Alox</u> | <u>1% Alox</u> |
| Current Production Aristowax: | | |
| Sample 1 | 8.6 | 5.5 |
| Sample 2 | 5.2 | 3.7 |
| Super Gem Amber C r | 2.4 | 1.3 |
| Glyco-Wax-A / 5%-Stearamide | 2.0 | 0.9 |

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Figure 11 shows the exudation at 60°C of Composition A pellets made with 1% Alox and various waxes, as a function of storage time in days. These particular tests were carried up to 35 days. The difference between values obtained with "Old Aristowax," part of a 500 lb. lot received in 1942 from the Union Oil Co., and with current Aristowaxes (the average results obtained with four different lots received during 1945) is very large - much greater than variations among current lots. Indeed the old sample is to be classed among the better, and the current samples among the poorer waxes from the standpoint of exudation. Among waxes procured during 1945, the one outstanding in excellence is Stanolind 170-75 (another Stanolind Wax, Stanolind 160-65 is, however, quite poor; the two should not be confused). On short-time storage, the Tank Bottom Wax, Super Gem Amber, also shows very low exudation, but beyond about two weeks it becomes poorer than Stanolind 170-75.

A few tests at 65°C, with waxes, in general, which were very good at 60°C, gave the following results (1% Alox 600 used in all cases):

| <u>Wax</u> | <u>Percent Wax Lost; 65°C, 5 days</u> |
|-----------------------------|---|
| Glyco-Wax A | 15.6 |
| Glyco-Wax A + 5% Stearamide | 12.3 |
| Super Gem Amber | 1.8 |
| Stanolind 170-75 | 0.3 |
| Current Aristowax | 11.5 |

Thus, even at this temperature, Stanolind 170-75 gives remarkably small exudation.

In the course of this report, it has been shown that the properties of the wax are of great importance for the final properties of Composition A; in particular, the oiling characteristics, pressing properties, chemical stability, and exudation depend strikingly upon properties of the desensitizing wax. It is imperative that waxes can be specified that will ensure to the finished products the properties desired. Moreover, waxes are used in other explosives, where other properties are, perhaps, required. Because of the magnitude of this problem - wax testing and wax specifications - it is being treated in a separate report, as OSRD No. 5633.

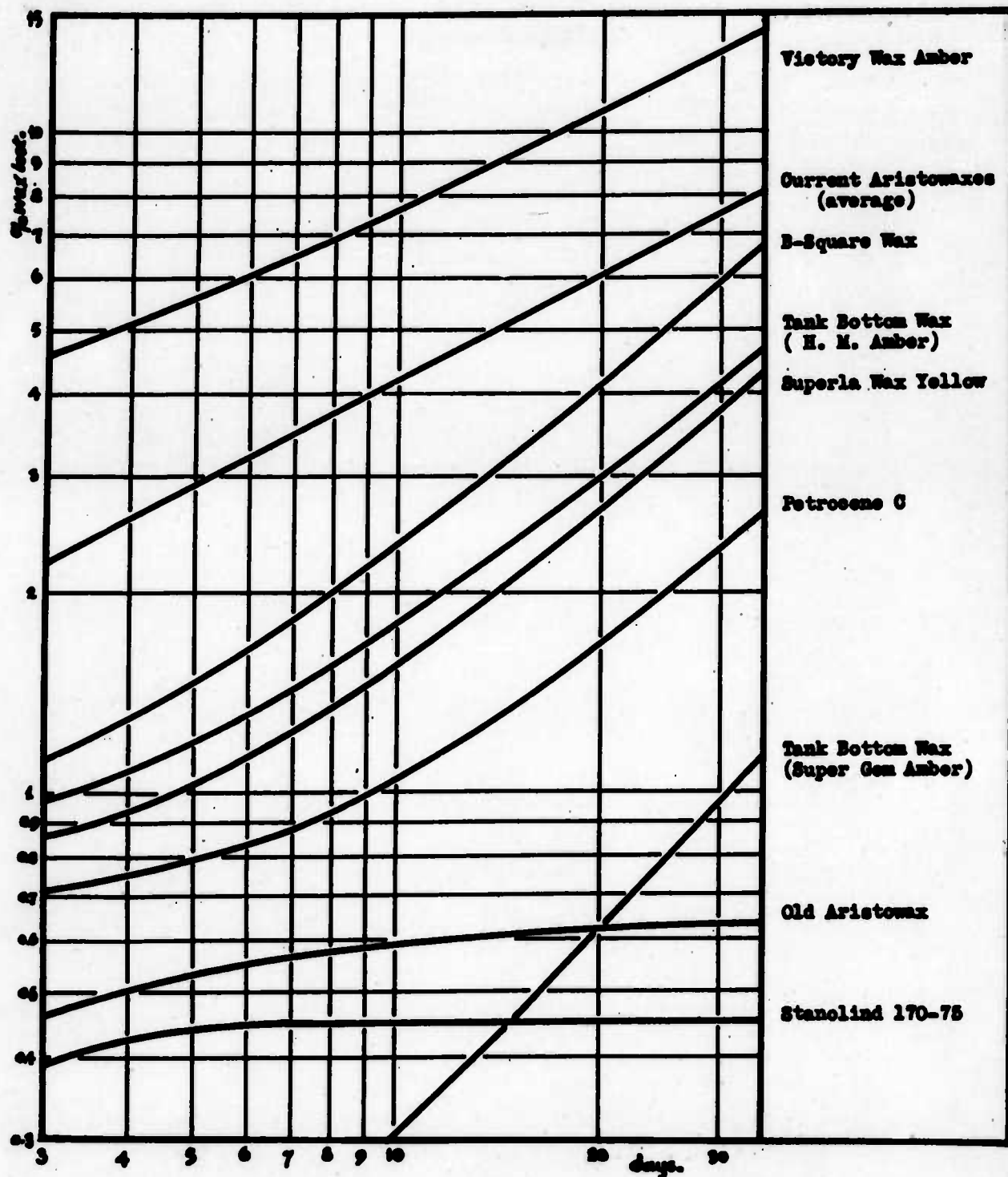


Figure 11. Loss in Weight of Composition A Pellets at 60° Centigrade as a Function of Storage Time in Days. Weight Loss Expressed as Percentage Wax Lost. One Percent Alex-600 Added to Each Wax.

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6. Variants of Composition A

(1) Aluminized Composition A

An RDX-Wax- Aluminum mixture called Aluminized Composition A has been described in Division 8 Interim Reports PT-21, p. 10, ff, and PT-22, p. 14. This material has the composition 73/9/18 RDX-Wax-Al, and has been satisfactorily made by oiling the 73 parts of RDX in slurry with 7 parts of wax, adding the aluminum when the waxed RDX is well grained, and finally, when the grains have been coated with aluminum, adding the remaining 2 parts of wax. The wet product is then dried like Composition A-3.

a. Sensitivity

The following Tables summarize the available sensitivity tests:

Drop-Weight Impact Tests

| <u>Explosive</u> | <u>50% point (cm)</u> | <u>50% point (cm)</u> |
|--------------------|-----------------------|-----------------------|
| | <u>Type 12</u> | <u>Type 13</u> |
| Composition A-3 | 80 | 218 |
| Aluminized Comp. A | 62 | 171 |
| Tritonal, 80/20 | 138 | 140 |

Bullet-Impact Tests; Cal. 0.30, Ball, m2; 1" pipe nipples

| <u>Explosive</u> | <u>No.</u> | <u>NE</u> | <u>EP</u> | <u>E</u> | <u>EC</u> |
|------------------------------|------------|-----------|-----------|----------|-----------|
| Comp. A-3 (d = 1.6) | 10 | 10 | -- | - | -- |
| Aluminized Comp. A (d = 1.6) | 11 | 10 | 1 | - | -- |
| Tritonal 85/15 (d = 1.6) | 10 | 10 | -- | - | -- |
| Comp. A-3 (d = 1.0) | 59 | 59 | -- | - | -- |
| Aluminized Comp. A (d = 1.1) | 10 | -- | 10 | - | -- |
| Tritonal 85/15 (d = 1.0) | 3 | -- | -- | - | 3 |

It is clear that each test reveals sensitization of Comp. A by the aluminum addition, but how the aluminized product compares, say, with Tritonal is not made clear. The

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bullet-test and the Type 13 Drop-Test suggest the Aluminized Comp. A to be less sensitive, while the Type 12 Drop-Test suggests that Aluminized Comp. A is more sensitive than Tritonal.

b. Performance

The following are results of laboratory measures of performance:

| <u>Explosive</u> | <u>Ballistic Mortar (T.V.)</u> | <u>Relative Brisance dens. (T.V.)</u> | <u>Relative Fragment Vel. dens. (T.V.)</u> | <u>Velocity of Detonation dens. V (m/sec)</u> |
|------------------|--|---|--|---|
| Comp. A-3 | 135 | 1.611 | 126 | 1.608 108 1.60 8200 |
| Alz'd Comp. A | 142 | 1.719 | 110 | 1.696 113 1.70 7800 |

c. Pressing

This material is very easy to press, giving less trouble with sticking and as good or better percent voids than Composition A under similar pressing conditions.

(2) 12% Composition A

Because of the failure of the 6" A.P. shell to pass the Navy Plate Test when loaded with Composition A, and more particularly because of the failure of the 5" - 38 A.A. shell, so loaded, to pass the mass detonation tests, we have been requested to make a brief study of a 88/12 RDX-Wax mixture.

a. Preparation

Such a mixture can ready be prepared like Composition A-3, by adding wax in water slurry. Somewhat larger grains are obtained unless conditions are altered to avoid this. Ultraviolet examination reveals good coatings. With this material there seems to be a tendency to lose even greater percentages of wax in hot storage than from regular Composition A-3; the use of waxes such as Stanolind 170-75 is especially desirable in making this product.

b. Pressing Properties

No extensive study of this matter has been made, but the following results were obtained using Stanolind 170-75 and 1% Alox 600.

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| <u>Pressure</u> <u>(psi)</u> | <u>12% Comp. A</u> <u>Density</u> | <u>9% Comp. A</u> <u>Density</u> |
|---------------------------------|--------------------------------------|-------------------------------------|
| 3000 | 1.45 | 1.44 |
| 8000 | 1.50 | 1.52 |
| 10000 | 1.54 | 1.57 |
| 15000 | 1.56 | 1.59 |

The differences are about accounted for by the difference in density of the mixtures.

c. Sensitivity

Since this mixture was requested in the hope that it might pass specific tests, those tests will decide whether its sensitivity is low enough. The Type 12 machine gives 50% Points in cm of 80 and 108 for 9 and 12% Comp. A, respectively.

d. Performance

Performance tests results available are summarized below:

| <u>Explosive</u> | <u>Ballistic</u> <u>Mortar</u> <u>(T.V.)</u> | <u>Relative</u> <u>Fragment:Velocity</u> <u>dens. (T.V.)</u> | <u>Velocity of</u> <u>Detonation</u> <u>dens. V(m/sec)</u> |
|-------------------|--|--|--|
| Composition A-3 | 135 | 1.608 108 | 1.60 8200 |
| 12% Composition A | 132 | 1.566 109 | 1.585 3240 |

In no case is the decline in performance great; indeed in fragment velocity and detonation velocity the reported values happen to be higher for the 12% Composition A.

(3) Composition A - KNO₃ Mixtures

Such mixtures have been studied as shell-fillers to give white burst. All work done in this laboratory has been collected in Division 8 Interim Report PT-36, p. 11, and need not be repeated here.

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ABSTRACT:

Preparation and properties of RDX-composition A are discussed, beginning with the preparation of composition A, followed by a thorough small-scale study of the pelleting properties of composition A. It is noted that of the possible methods of coating RDX with wax, the slurry method is deemed best; and on the study of pelleting properties it is shown that the pellet-density may be represented basically as a function of the wax hardness and the pressure. The explosive properties of composition A is next discussed and, because of its constitution and mechanical properties, it is not easy to compare, in sensitivity, with pure compound explosive materials. Finally such variants of composition A as aluminized composition A, 12% composition A, etc., are briefly discussed.

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